Recovery of failed solid-state anaerobic digesters

Liangcheng Yang¹, Xumeng Ge², Yebo Li^{2*}

¹ Department of Health Sciences, Illinois State University, 324 Felmley Hall, Normal, IL 61790,

USA. Phone: +1 309 438 7133. E-mail lyang@ilstu.edu

² Department of Food, Agricultural and Biological Engineering, The Ohio State University/Ohio Agricultural Research and Development Center, 1680 Madison Ave., Wooster, OH 44691-4096,

USA

* Corresponding author. Phone: +1 330 263 3855. E-mail li.851@osu.edu

Abstract

This study examined the performance of three methods for recovering failed solid-state anaerobic digesters. The 9-L digesters, which were fed with corn stover, failed at a feedstock/inoculum (F/I) ratio of 10 with negligible methane yields. To recover the system, inoculum was added to bring the F/I ratio to 4. Inoculum was either added to the top of a failed digester, injected into it, or well-mixed with the existing feedstock. Digesters using top-addition and injection methods quickly resumed and achieved peak yields in 10 days, while digesters using well-mixed method recovered slowly but showed 50% higher peak yields. Overall, these methods recovered 30-40% methane from failed digesters. The well-mixed method showed the highest methane yield, followed by the injection and top-addition methods. Recovered digesters outperformed digesters had a constant F/I ratio of 4. Slow mass transfer and slow growth of microbes were believed to be the major limiting factors for recovery.

Keywords: digester failure, recovery, mass transfer, solid-state anaerobic digestion

1. Introduction

There has been growing interest in developing solid-state anaerobic digestion (SS-AD), which can be used with solid contents greater than 15%, due to its high organic loading rate, small digester volume, and high volumetric methane production (Li et al., 2011). A recent study showed that for new digestion projects in Europe, SS-AD has been preferred over conventional low solid anaerobic digestion (Karthikeyan & Visvanathan, 2013). A major challenge of SS-AD is low mass transfer efficiency between inoculum and feedstock, due to the high solids content, which leads to a series of problems such as accumulation of inhibitors, imbalance of nutrients, and low methane yield (Fagbohunge et al., 2015; Yang et al., 2014). It was found that the mass diffusion rate of digesting material decreased by 3.7 times when the solid content increased from 8% to 25% (Bollon et al., 2011). The high solid content of 30–35% may also hinder gas-liquid transfer, thus leading to accumulation of carbon dioxide and hydrogen that may inhibit microbes. These potential problems contribute to instability in SS-AD systems, which may eventually cause digester failure (Graef & Andrews, 1974).

To date, most studies have focused on optimization of digester operation to minimize the chance of failure, little work has been done to recover failed digesters (Li et al., 2014; Zhu et al., 2014). Efforts such as recirculation of biogas and leachate back to digester, premixing of inoculum and feedstock before digestion, and mechanical mixing of inoculum and feedstock during digestion, have been made to improve mass transfer during SS-AD (Karagiannidis & Perkoulidis, 2009; Verma, 2002; Zhu et al., 2014). However, the SS-AD digestion process involves many factors and so far there is no single effective solution to prevent failure. Li et al. (2014) tested a variety of operating parameters but showed that no single indicator was universally valid as an early

70 warning for digestion failure. Also, due to high solid content, feedstock in failed digesters cannot
71 be pumped out, but has to be removed by using toe tip buckets, which is a slow and expensive
72 process. Given the possibility and economic cost of digester failure, an efficient and cost-
73 effective recovery plan is needed, especially for large scale solid-state anaerobic digesters.

74
75 Recovery within the reactor of a failed digester would be the preferred solution. Theoretically,
76 adding a large amount of inoculum into a failed digester should resume methane production.
77 Inoculum addition has been suggested as method to provide active digestion microbes and dilute
78 inhibitors (e.g. volatile fatty acids), therefore, helps recovering failed digesters (Aboudi et al.,
79 2016). In a previous study, directly adding extra inoculum to the top of failed digesters without
80 further mixing resumed biogas production (Zhu et al., 2014). Besides inoculum, other agents
81 such as alkaline has been applied (Liew et al., 2011). For low solid continuous digesters,
82 recovery of failed digesters may be accomplished by ceasing feeding or re-inoculation with a
83 well-adapted inoculum (Aboudi et al., 2016). The exchange of digesting sludge between serial
84 reactors has also been demonstrated as an effective approach for transferring excess volatile fatty
85 acids from the first reactor to other reactors, thereby recovering the failed digester (Kafle & Kim,
86 2011).

87
88 Two concerns for recovery of solid-state anaerobic digesters are the availability of space within
89 the digester and the energy required for mixing. The available space determines the maximum
90 amount of inoculum or other recovery agents that can be added into a failed digester; while the
91 method for mixing the recovery agent with existing digestion feedstock can affect energy
92 demand and recovery outcomes. Previous modeling studies suggested that uniform inoculum

distribution via complete mixing would outperform non-uniform inoculum distribution (Vavilin et al., 2008). However, well-mixing of the recovery agent with the existing feedstock can be a practical challenge due to high solids content and high viscosity. Thus for the best results, the recovery agent and feedstock need to be mixed outside of the digester. Well-mixing also requires high energy inputs.

In this study, the effects of three methods of adding inoculum to recover a failed digester were tested: top-addition, injection, and well-mixing. Top-addition refers to adding recovery agent (in this test, was inoculum) directly onto the top of the feedstock in the failed digester, injection refers to injecting the inoculum into the central area of the failed digester, and well-mixing refers to thoroughly mixing the added inoculum with the feedstock. A limited amount of inoculum was used for recovery to avoid space concerns. The objectives of this study were to: (1) examine the performance of the three recovery methods in recovering failed solid-state anaerobic digesters, and (2) explore limiting factors during recovery.

2. Methods and materials

2.1 Feedstock and inoculum

Corn stover collected from a research farm at the Ohio Agricultural Research and Development Center (OARDC) in Wooster, OH, USA, in January 2013 was used as the feedstock in this study. Corn stover was air dried to less than 15% moisture content, and then ground to pass a 6 mm sieve (Mighty Mac, MacKissic Inc., Parker Ford, PA, USA). Effluent taken from a mesophilic liquid anaerobic digester (fed with municipal sewage sludge and food wastes, operated by *quasar energy group* in Zanesville, OH, USA) was used as the inoculum. The inoculum was activated in

a 37°C incubation room for one week prior to use. Characteristics of the feedstock and inoculum are shown in Table 1.

2.2 Anaerobic digestion

The SS-AD recovery test was carried out in 9-L plastic digesters (ID: 15 cm, Height: 52 cm, Fig. 1) without leachate or biogas recirculation in a 37°C incubation room. The corn stover (400 g) was mixed with inoculum and then loaded into the digesters, with an effective height of about 30 cm (effective volume of 5.3 L). The mixing ratio was based on the volatile solid content of the feedstock and inoculum. The initial F/I ratio (feedstock/inoculum) was 10, and the initial total solids (TS) content was 32%. With the high F/I ratio and TS, digesters would most likely fail due to insufficient inoculum supply and retarded mass transfer (Sheets et al., 2015; Zhu et al., 2014). Recovery of failed digesters was carried out on day 20, by adding inoculum into the digester to lower the F/I ratio to 4 and decrease TS to 20 (Table 2). As shown in Figure 1, the inoculum was added in one of three ways: (1) Inoculum was added directly onto the top of the feedstock in the digester (top-addition), so that the inoculum would gradually infiltrate the feedstock. (2) A plastic rod was used to create a 2.54 cm wide, 20 cm deep hole in the center of the failed digester, and then inoculum was injected into the hole (injection). The inoculum was expected to infiltrate into the surrounding annular space and bottom area. (3) The feedstock was removed and completely mixed with the added inoculum, and then re-loaded into the digester (well-mixing). These three methods were used to mimic possible recovery practices for large-scale digesters. For each digester, a 10-L Tedlar gas bag (CEL Scientific, Santa Fe Springs, CA, USA) was attached to collect biogas every 2–5 days during the 5-month experimental period.

Besides the digesters used for the recovery tests, several other digesters were used as the controls (Table 2). Two 1-L digesters (control 1) were operated at a low F/I ratio of 0.5 and a low total solids content of 9.5%, and were on a shaker during the entire digestion process, which was expected to generate close to maximum biogas yield. Two 1-L digesters (control 2) were operated at an F/I ratio of 4 and total solids content of 20; and one 9-L digester (control 3) was operated at an F/I ratio of 10 and was not recovered.

2.3 Analytical methods

The volume of biogas was measured with a drum-type gas meter (Ritter, Bochum, Germany) and its composition (CH_4 , CO_2 , N_2 , and O_2) was analyzed with a gas chromatograph (Agilent, HP 6890, Wilmington, DE, USA) equipped with a $30\text{ m} \times 0.53\text{ mm} \times 10\text{ }\mu\text{m}$ Rt® -Alumina Bond/KCl deactivation column and a thermal conductivity detector. Helium gas was used as a carrier gas at a flow rate of 5.2 mL/min. The temperature of the detector was maintained at 200°C, while the initial temperature of the oven was 40°C and then increased to 60°C within one minute. The TS, VS, pH, and alkalinity of the feedstock and inoculum were measured based on the Standard Methods Examination of Water and Wastewater (APHA, 2005). Specifically, a 5 g sample was diluted with 50 ml of deionized (DI) water, and then the pH and alkalinity were measured using an auto-titrating pH meter (Mettler Toledo, DL22 Food & Beverage Analyzer, Columbus, OH, USA). Total carbon and total nitrogen contents were determined using an elemental analyzer (Elementar Vario Max CNS, Elementar Americas, Mt. Laurel, NJ, USA). Volatile fatty acids (VFAs), which include propionic, acetic, isovaleric, butyric, isobutyric, and valeric acids, were analyzed using a gas chromatograph (Shimadzu, 2010PLUS, Columbia, MD, USA) equipped with a $30\text{ m} \times 0.32\text{ mm} \times 0.5\text{ }\mu\text{m}$ Stabilwax® polar phase column and a flame

ionization detector according to a method described previously (Shi et al., 2013). Monomeric sugars (glucose, xylose, galactose, arabinose, and mannose) were analyzed using high-performance liquid chromatography (Shimadzu, LC-20AB, Columbia, MD, USA) equipped with a Biorad Aminex HPX-87P column and a refractive index detector. HPLC grade water was used as the mobile phase at a flow rate of 0.3 mL/min, and temperatures of the column and detector were maintained at 60°C and 55°C, respectively. Statistical analysis of the data was conducted using SigmaPlot and R Studio software. Averages and standard errors are reported.

3. Results and discussion

3.1 Performance of the controls

As shown in Fig. 2, at a low F/I ratio of 0.5 and low TS of 9.5 with continuous shaking (control 1), the daily methane yield peak value (13.3 L/kg-VS.day) was reached on day 13 and methane content stabilized on day 19. The accumulative methane yield was 330.5 L/kg-VS (Fig. 3). This result is considered as the maximum achievable methane yield, and is close to the results reported by other researchers (Xu et al., 2013). Control 2 and 3 digesters (Table 2), however, quickly failed, producing only 2.4 and 0.4 L/kg-VS of methane, respectively (Fig. 3). Failure at an F/I ratio of 10 was expected based on previous studies (Xu & Li, 2012); while failure at F/I ratio of 4 reflects the instability of SS-AD as discussed in the introduction.

3.2 Recovery performance

For all treatments, biogas yields were lower than 0.1 L/kg-VS during the first 20 days and, therefore, were considered as failed. The recovery step was carried out on day 20 and

significantly increased methane yield thereafter (Fig. 4). Different patterns in methane yield and methane content were observed for the three recovery methods.

With the top-addition method, daily methane yield reached the stable phase 10 days after recovery and lasted for 1 month, and then gradually decreased (Fig. 4A). The peak daily methane yield (1.01 L/kg-VS.day) was much lower than that obtained from recovered digesters using other methods (Fig. 4B and 4C), and also much lower than the control 1 digesters. Based on methane content, the digesters also took a longer time (~25 days) to reach the stable phase (Fig. 4D). The low daily methane yield and long time for stabilization could have been caused by slow infiltration of inoculum. The inoculum was sticky and had a moderate solid content of 8.06% (Table 1), and thus had limited mobility. Even though it was hard to confirm visually, it is likely that the added inoculum did not reach the bottom of digesters by the end of the recovery test, as evidenced by differences in color and moisture content. The bottom layer of the feedstock had a lighter color and lower moisture content compared to the top layer where the inoculum was added. Slow infiltration of inoculum would have reduced access to feedstock for methane production. In addition, aerobic microbes may have been enabled, resulting in the production of other gases, which contributed to the low methane content and yield at the beginning of recovery.

With the injection method, the daily methane yield reached the first peak 10 days after recovery was initiated, and then obtained a second peak 33 days after the first peak (Fig. 4B). Compared to the top-addition method, the daily methane yield with injection recovery was about two times higher, which may be because of the larger contact surface area between the feedstock and added inoculum, as the inoculum was introduced to the surrounding annular space and bottom area (Fig.

1). Possibly, the first peak was generated from the immediately available feedstock in the central and the bottom area where the inoculum was injected; while the second peak may have been caused by the slow infiltration of inoculum. The methane content in biogas slowly increased to 55% 32 days after recovery and then remained constant (Fig. 4E). The slow increase of methane content was similar to that observed from the digesters that used the top-addition method, and was very likely caused by the slow infiltration of inoculum. Both methane yield and methane content were highly dynamic as indicated by the large standard errors (Fig. 4B and 4E). These large errors could have been caused by the uneven diffusion and distribution of inoculum in the digesters.

With the well-mixing method, the daily methane yield slowly increased to 2.49 L/kg-VS 54 days after recovery was initiated (Fig. 4C). The peak value was 50% higher than that obtained from the digesters using the injection method, and 150% higher than those using the top-addition method. However, it took a much longer time than the other two methods to reach peak yields, and dropped quickly after that. The overall profile of daily methane yield using the well-mixing method was similar to the control 1 digesters (Fig. 2), but with a longer progressing time and a lower yield. The methane content quickly jumped to 53% in 16 days after recovery, which was shorter than the other two recovery methods, but again similar to the control 1 digesters. Interesting differences were observed when comparing the digesters using the well-mixed method to control 2 digesters. Control 2 digesters, which were well-mixed initially, had a constant F/I ratio of 4 (Table 2), and failed quickly. While the digesters using the well-mixing recovery method, which had an initial F/I ratio of 10 that was decreased to 4, succeeded in producing biogas. The failure of control 2 digesters likely was caused by the over accumulation

of digestion intermediates, e.g. volatile fatty acids and free ammonia, which inhibited methanogens (Yang et al., 2014). While for the recovery test, the inoculum added initially (F/I ratio of 10), although not able to produce biogas, should have created a suitable digestion environment in which volatile fatty acids were partially consumed by aerobic or anaerobic microbes, increasing the overall alkalinity. Therefore, when inoculum was added for recovery, inhibition on methanogens may not have been as extensive as in the control 2 digesters, so that methanogens grew slowly as indicated by the long lag phase as shown in Fig. 4C.

The cumulative methane yields from all controls and treatments are shown in Fig. 3. The methane yields from controls 2 and 3 were negligible. The Student's t-test results showed that all three recovery methods significantly improved methane yield compared to the control 3 digesters ($p < 0.01$). Based on this test only, the well-mixing method recovered the highest amount of methane, followed by the injection and top-addition methods. The methane yields from the recovered digesters were equal to 30-40% of that produced from control 1 digesters, which had a low F/I ratio of 0.5 and a low TS content of 9.5 and were shaken continuously. If more inoculum was used for recovery, it is highly likely that more methane could be produced.

3.3 Limiting factors of recovery

Based on the pattern of methane production after recovery, the slow infiltration of inoculum seemed to be a major obstacle for the top-addition and injection methods. In another words, the limiting factor was the slow mass transfer. Methods such as multiple layer addition and multiple injection holes would increase contact between inoculum and feedstock, and, therefore, may improve methane yield. For the well-mixing recovery method, the contact between inoculum and

feedstock was not a problem, but the presence of inhibitory agents and the slow growth of digestion microbes were. One possible way to solve these problems is to increase the amount of inoculum for recovery, which would dilute inhibitors and provide sufficient digestion microbes.

Besides methane yield, selection of a recovery method would also be affected by other factors, such as energy, space outside the digester, and equipment requirements as well as available space inside the digester. Top-addition and injection methods, even with multiple layers or holes, are relatively simple; whereas, the well-mixing method is energy consuming and usually needs extra space as it can be difficult to mix inoculum and feedstock inside of a failed digester due to the high solids content. All three recovery methods are affected by the availability of space in the digester. In this test, the amount of inoculum added for recovery (to decrease F/I ratio 4) was marginal but was still able to resume biogas production. Therefore, it is envisaged that recovery of failed digesters can be feasible for large scale solid-state anaerobic digesters.

4. Conclusion

All three recovery methods resulted in resumed methane production at an F/I ratio of 4. Overall methane yields from recovered digesters were equal to 30-40% of that obtained from the control digesters with a low F/I ratio of 0.5, and outperformed control digesters that had a constant F/I ratio of 4. The well-mixing method showed the highest methane yield, followed by injection and top-addition methods. Slow mass transfer could be the limiting factor for top-addition and injection methods, while slow growth of digestion microbes may have obstructed well-mixed digesters.

Acknowledgement

This project was funded by USDA NIFA Biomass Research and Development Initiative Program (Award No. 2012-10008-20302). The authors wish to thank Mrs. Mary Wicks (Department of Food, Agricultural and Biological Engineering, OSU) for critical review.

References

- Aboudi, K., Álvarez-Gallego, C.J., Romero-García, L.I.R. 2016. Biomethanization of sugar beet byproduct by semi-continuous single digestion and co-digestion with cow manure. *Biores. Tech.*, **200**, 311-319.
- APHA. 2005. Standard Methods for the Examination of Water and Wastewater. 21st Ed., American Public Health Association. Washington, D.C.
- Bollon, J., Le-Hyarc, R., Benbelkacem, H., Buffiere, P. 2011. Development of a kinetic model for anaerobic dry digestion processes: Focus on acetate degradation and moisture content. *Biochem. Eng. J.*, **56**(3), 212-218.
- Fagbohunge, M.O., Dodda, I.C., Herbertb, B.M.J., Li, H., Ricketts, L., Semple, K.T. 2015. High solid anaerobic digestion: Operational challenges and possibilities. *Environ. Technol. Innov.*, **4**, 268-284.
- Graef, S.P., Andrews, J.F. 1974. Stability and control of anaerobic digestion. *Water Pollut. Control Federat.*, **46**(4), 666-683.
- Kafle, G.K., Kim, S.H. 2011. Sludge exchange process on two serial CSTRs anaerobic digestions: Process failure and recovery. *Biores. Tech.*, **102**(13), 6815-6822.

296 Karagiannidis, A., Perkoulidis, G. 2009. A multi-criteria ranking of different technologies for the
 297 anaerobic digestion for energy recovery of the organic fraction of municipal solid wastes.
 298 *Biores. Technol.*, **100**(8), 2355-2360.

299 Karthikeyan, O.P., Visvanathan, C. 2013. Bio-energy recovery from high-solid organic
 300 substrates by dry anaerobic bio-conversion processes: A review. *Rev. Environ. Sci.*
 301 *Biotechnol.*, **12**(3), 257-284.

302 Li, L., He, Q., Wei, Y., He, Q., Peng, X. 2014. Early warning indicators for monitoring the
 303 process failure of anaerobic digestion system of food waste. *Biores. Tech.*, **171**, 491-494.

304 Li, Y., Zhu, J., Wan, C., Park, S.Y. 2011. Solid-state anaerobic digestion of corn stover for
 305 biogas production. *Trans. ASABE*, **54**(4), 1415-1421.

306 Liew, L.N., Shi, J., Li, Y. 2011. Enhancing the solid-state anaerobic digestion of fallen leaves
 307 through simultaneous alkaline treatment. *Biores. Tech.*, **102**(19), 8828-8834.

308 Sheets, J.P., Yang, L., Ge, X., Wang, Z., Li, Y. 2015. Beyond land application: Emerging
 309 technologies for the treatment and reuse of anaerobically digested agricultural and food
 310 waste. *Waste Manag.*, **44**, 94-115.

311 Shi, J., Wang, Z.J., Stiverson, J.A., Yu, Z.T., Li, Y.B. 2013. Reactor performance and microbial
 312 community dynamics during solid-state anaerobic digestion of corn stover at mesophilic
 313 and thermophilic conditions. *Biores. Tech.*, **136**, 574-581.

314 Vavilin, V.A., Fernandez, B., Palatsi, J., Flotats, X. 2008. Hydrolysis kinetics in anaerobic
 315 degradation of particulate organic material: An overview. *Waste Manag.*, **28**(6), 941-953.

316 Verma, S. 2002. Anaerobic digestion of biodegradable organics in municipal solid wastes. . in:
 317 *Department of Earth and Environmental Engineering, Fu Foundation School of*
 318 *Engineering and Applied Science, Columbia University.*

- Xu, F., Li, Y. 2012. Solid-state co-digestion of expired dog food and corn stover for methane production. *Biores. Technol.*, **118**, 219-226.
- Xu, F.Q., Shi, J., Lv, W., Yu, Z.T., Li, Y.B. 2013. Comparison of different liquid anaerobic digestion effluents as inocula and nitrogen sources for solid-state batch anaerobic digestion of corn stover. *Waste Manag.*, **33**(1), 26-32.
- Yang, L., Xu, F., Ge, X., Li, Y. 2014. Challenges and strategies for solid-state anaerobic digestion of lignocellulosic biomass. *Renew. Sustain. Energy Rev.*, **44**, 824-834.
- Zhu, J., Yang, L., Li, Y. 2014. Comparison of premixing methods for solid-state anaerobic digestion of corn stover. *Biores. Tech.*, **175**, 430-435.

342
343
344
345
346

347

348

349

350

351

Table 1 Properties of feedstock and inoculum

Parameters	Feedstock	Inoculum
TS, %	89.14±0.43	8.06±0.01
VS, %	84.05±0.61	3.94±0.01
TC, %	43.40±0.23	2.60±0.07
TN, %	0.60±0.00	0.32±0.01
C/N ratio	71.86±0.75	8.19±0.59
pH	6.41±0.02	8.00±0.01
Alkalinity, g-CaCO ₃ /kg	0.19±0.00	10.76±0.21
VFAs, g/kg	0.27±0.02	0.93±0.06
Cellulose ^a , %	34.40±0.35	1.75±0.13
Hemicellulose ^a , %	20.61±0.05	1.01±0.00

TS=total solids, VS=volatile solids, TC=total carbon, TN=total nitrogen, C/N= carbon to nitrogen, VFAs=volatile fatty acids. ^a based on TS. Others are based on total weight. Average ± S.E., n=2.

352

Table 2 Operating conditions of digesters

Conditions	# of digesters	Digester Vol (L)	Initial condition		After recovery		Operation time (months)
			F/I	TS	F/I	TS	
Top-addition	2	9	10	32	4	20	5
Injection	2	9	10	32	4	20	5
Well-mixing	2	9	10	32	4	20	5
Control 1	2	1	0.5	9.5	No recovery		2
Control 2	2	1	4	20	No recovery		2
Control 3	1	9	10	32	No recovery		5

353

354

355

356

357

358

359
360
361
362
363
364
365
366
367
368
369
370
371
372
373
374
375

Fig. 1 SS-AD digester recovery methods. a): top-addition, b): injection, and c): well-mixing.

Fig. 2 Daily methane yield and methane content of digesters with F/I=0.5 (control 1 digesters).

Solid line: daily methane yield; dashed line: methane content.

392

393

394 Fig. 3 Comparison of cumulative methane yield from the control and recovered digesters.

395

396

397

Fig. 4 Daily methane yield (A, B, C) and methane content (D, E, F) from recovered digesters.

The recovery process was initiated on day 20.