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<https://doi.org/10.15760/honors.368>

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BENCHTOP MINIMAL-INTERVENTION ANAEROBIC DIGESTION OF
VEGETARIAN FOOD WASTE FOR pH AND METHANE PRODUCTION:
CONCEIVABILITY AND CONTROL STUDY

BY

Emily Heleva-Ponaski

A thesis submitted in partial fulfillment
of the requirement for the degree of

BACHELOR OF SCIENCE WITH DEPARTMENTAL HONORS
IN
CIVIL AND ENVIRONMENTAL ENGINEERING

Thesis Advisor:

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ACKNOWLEDGEMENTS

I would like to thank the Maseeh College of Computer Science and Engineering for funding our project through the awarding of an Innovation Grant. Thanks to Dr. William Fish for all of his advice and expertise, and letting us make his lab the smelliest place in the building.

I also wish to thank the PSU staff who helped us with our original, large-scale digester project that never came to fruit: Tom Bennett, Tom Szymoniak, Bradley Melaugh, Anthony Hair, and the entire PSU Environmental Club. Hopefully someday the orchard will have a food waste digester.

Lastly, I'd like to thank my research partner Leland Cornwallis Scatlebury for working numerous hours with me on this project through grueling days, awful smells, moments of frustration and even a broken windshield. There would have been many more tears and less laughter without you.

ABSTRACT

Cities around the world transport large quantities of waste to landfills at a great expense to their residents, infrastructures, and environments. The objective of this study was to run an anaerobic food waste digester with minimal interference or maintenance. One specific goal of this research was to evaluate the relationship between anaerobic food waste and pH. Two benchtop digesters were started with vegetarian food waste collected from the Portland State University campus. Measurements were collected over the course of the digestion process. Due to low pH and lack of biogas production, the digesters were buffered with sodium carbonate and seeded with wastewater digestate. Post recovery, the solids content decreased and methane production began though the process was never fully optimized. The lack of digester performance is thought to be due in part to low operation temperature.

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INTRODUCTION

Cities around the world transport large quantities of waste to landfills at a great expense to their residents, infrastructures, and environments. In America, families discard nearly 25% (by mass) of the food they purchase (Gunders, 2012), not including inedible portions, which in addition to commercial food waste becomes a sizeable portion of total waste. At 6 million tons, food constitutes 15.5% of California's waste (CIWMB, 2008). Food waste then decomposes resulting in up to 23% of America's methane emissions (Gunders, 2012).

Anaerobic digestion is a well-established method for breaking down solids into useful byproducts of nutrient-rich liquid fertilizer and methane gas (Gray et al., 2008). While methane is a powerful greenhouse gas, properly collected and stored it can be a useful fuel and a source of renewable energy. While relatively uncommon in the United States, high-solids food waste digestion is becoming increasingly popular in Asia and Europe (De Baere L., 2000). Thus potential exists for America to reduce greenhouse gas emissions and landfill-bound food waste while generating electricity.

The digestion process consists of four main stages, hydrolysis, fermentation, acetogenesis and methanogenesis as (Figure 1). During hydrolysis, complex molecules like proteins, lipids, and carbohydrates are broken down into simpler molecules by extracellular enzymes (Li et al., 2010).

Figure 1. Anaerobic digestion process (Droste 1997).

Bacteria then ferment these amino acids, fatty acids, and sugars. The products of fermentation vary depending on the types of bacteria present (which is in turn dependent on the pH and temperature). Fermentation produces some amount of acetate, carbon dioxide, and hydrogen, but primarily creates volatile fatty acids used as a substrate during acetogenesis. Acetogenesis continues the creation of acetic acid, carbon dioxide, and hydrogen, which are the primary substrates for methane production (Li et al., 2010). The reaction for glucose (as an example substrate) conversion to acetic acid is:

(Thompson, 2008). This acid-forming stage is generally optimized around a pH of 4.5-6 (Demirel & Yenigün, 2002), contrasting with common digester operation at a neutral pH.

Methanogenesis is carried out by Achaea, single-celled organisms in their own kingdom separate from bacteria and eukaryotes. These methanogens primarily use acetic acid to produce methane in the overall reaction:

But methanogens can use a variety of substrates to produce methane, such as hydrogen and carbon dioxide:

(Droste, 1996 and Thompson, 2008).

Common food waste, after processing (e.g. blending) has a greater solids content than traditional wastewater digester feedstock, at 15% or higher (Li et al., 2011). Food waste also has a much higher COD (Min et al., 2005), which indicates a greater potential for producing methane (Droste, 1996). Anaerobic digestion of solid waste of all sources (e.g. food, manure) is seen as an important way of treating waste and producing energy in developing countries (Müller, 2007). Operations of any size provide communities with the opportunity to produce fuel or electricity locally. American communities could also localize their food waste disposal, reducing transportation costs and total waste.

Stability of the anaerobic digestion process can be difficult to start and maintain, largely due to the diverse needs and sensitivities of the involved organisms (Chen et al., 2007). Digesters generally require a pH held at neutral and a dedicated heat source. Food waste-only digesters can be especially difficult, lacking the diverse quantity of bacteria present in manure and with more material to be broken down.

Due to the great potential of the technology, a desire exists to create small scale, minimal maintenance anaerobic food waste digesters. Such digesters could use solar radiation as a heat source and ideally would require no pH control or bacterial seeding. The objective of this study was to run an anaerobic food waste digester with minimal interference or maintenance. Additionally, the amount of substrate processing necessary

was to be evaluated, comparing chopped waste and blended waste. Upon failure of a digester (defined by low pH, lack of solids reduction, and lack of methane production) appropriate steps were to be taken to recover it, through sodium carbonate (Na_2CO_3) pH control, heating, or reseeded. The high coffee ground content of our food waste supply indicated likely pH control at the very least would be necessary (Kozuchowska & Evison, 1995).

One specific goal of this research was to evaluate the relationship between anaerobic food waste and pH. More particularly, concentrating on the type of substrate and their contribution to the system's pH. Previous studies have shown pH control was necessary for food waste digestion and methane production (Zhang et al, 2010). Although pH control seems inevitable, the main goal is to provide the most minimal type of intervention.

METHODS

Experimental Apparatus

The experimental apparatuses shown in Figure 2 and 3 consisted of a hard plastic container (pre recovery)/a flexible plastic cube container (post recovery) approximately half full with substrate. A balloon was attached to a valued lid to allow for gas collection.

Figure 2. Hard plastic container (pre recovery).

Figure 3. Flexible cube container (post recovery).

Initial Apparatus

A hard, translucent three-gallon container was filled halfway with food waste and sealed. A 36-inch balloon was attached to a hole approximately one centimeter in diameter near the top of the container to allow for gas collection and expansion. Initially the digesters were placed under the laboratory hood and wrapped with insulation. After a week, the digesters were moved to a table approximately one foot away from a window in the lab. The window was west facing so the digesters received afternoon sunlight. Since the containers were not completely full, the systems were not initially anaerobic.

Recovery Apparatus

Digester substrate was transferred to flexible transparent plastic cube containers. Each container had a valve opening that had a balloon attached to it for gas collection. The valve and the container flexibility allowed for the creation of an anaerobic environment inside the digester. The digesters were buffered and seeded during this container transition (see Digester seeding and recovery) and again placed in front of the window.

Experimental Procedure

Substrate Collection and Processing

Chunky contained two gallons of vegetarian waste from a campus restaurant kitchen, one gallon of vegetarian wood waste from Emily's kitchen, approximately half a cup of almond butter, and three liters of tap water. The food waste was chopped into half-inch cubes and mixed by shaking the whole container. Water was added 18 days later to Chunky.

Skinny contained approximately three gallons of food waste from the Smith Memorial Student Union (Portland State University campus) compost bin and three liters of tap water. The bin was not classified as vegetarian, but attempts were made to only select vegetarian items. About 50 percent of the substrate was coffee grounds and the remaining portion was dominated by orange and banana peels. Seventy five percent of the substrate was blended before being added to the digester. Skinny was started 18 days after Chunk, at the same time that water was added to Chunky.

Digester Startup and Operation

After Skinny's 18 day-late startup, caution was taken to handle both digesters in the same way in regards to sampling, seeding, buffering, location and mixing. After 19 days of tandem operation, both digesters had low, acidic pHs and were no longer producing gas. Titrations were performed on samples on each substrate to determine the amount of sodium carbonate (Na_2CO_3) necessary to recover the system to the desired pH (pH ~7).

Digester Seeding and Recovery

Each digester was transferred into a 20-liter cube container. The amount of sodium carbonate (solid) added each digester was informed by the titrations that had been previously performed. After the addition, each digester was mixed for approximately two minutes. Additional sodium carbonate was added to each until desired pH was reached. Air was then pushed out of the valve and sealed to create an anaerobic environment. After 48 hours, pH measurements were again taken to verify the pH had stabilized. The digesters were then seeded with two liters of digested sludge from Clean Water Services Durham wastewater treatment facility in Tigard, Oregon. Both digesters were again

mixed and pH was recorded. At this time, balloons were attached to the valves for gas collection.

Sampling and Analyses

For the titrations, 120mL of substrate was placed in a breaker. An initial pH reading was taken. Sodium carbonate (solid) was added in 0.5gram increments and mixed vigorously until the change in pH was less than 0.1. The pH measurements that were taken at each increment were plotted against the total base addition. This graph was used to calculate the amount of base needed to bring the whole system up to the desired pH.

To evaluate the influence of the carbonate system on the pH, samples of 200mL from each digester were poured into 500mL beakers. Initial pH measurements were taken. The sample was stirred and sat for 24 hours uncovered in the lab. After this time period, another pH sample was taken.

Total solids samples and pH measurements were collected on an average of every 7.4 days. In order to get a representative sample, approximately 200mL of substrate was poured into a beaker and mixed. Three to five samples were then processed in accordance with the procedures described in the Methods 1684 from the U.S Environmental Protection Agency. A pH probe was used to measure the pH. Volatile solids samples were taken once before the digester recovery and again after the digesters had consistently been producing gas. Triplicates were taken of each digester both times and processed in accordance with the procedures described in the Methods 1684 from the U.S Environmental Protection Agency.

Gas data was collected on three dates selected based on the pressure buildup within the digesters. Gas volumes produced were estimated based on modeling the air-

filled portion of the digester containers as rectangular boxes and the (never fully filled) balloons as cylinders. Volumes were recorded just prior to releasing the gas for methane (CH₄) concentration estimation. The gas was released from the digesters and run past a Hanwei Electronics MQ-4 methane gas sensor under a laboratory fume hood. The resistance of the sensor was read by an Arduino Uno hooked up to a laptop computer continually recording the values. Due to reaching the sensor detection limit of 10,000 ppm CH₄, this data was used only qualitatively and comparatively.

RESULTS

pH Control

Carbonate System Test

The pH of Chunky and Skinny were, respectively. After being stirred and left uncovered for 24 hours, the pH of both samples changed less than .

Titration

Chunky had a pH of 4.44 at the beginning of the titration and stabilized around 9. The titration was performed on 120mL of substrate to which half gram increments of sodium carbonate were added to reach the plateau point. This data was use to create a titration curve (Appendix A). From this curve we calculated that Chunky would need 136 grams of sodium carbonate to reach a pH ~7. After the initial addition of base, the pH immediately rose to 6.66, but dropped to 6.54 after two hours. More base was added (25.9 grams) raising the pH to 7.58. After 24 hours, the pH of Chunky stabilized at 7.05.

Skinny had a pH of 3.62 at the beginning of the titration and stabilized close to 10. The titration was performed on 120mL of substrate to which half gram increments of sodium carbonate were added to reach the plateau point. From this curve we calculated

that Skinny would need 189 grams of sodium carbonate to reach a pH ~7. After the initial addition of base, the pH immediately rose to 6.31, but dropped to 6.00 after two hours. More base was added (75.1 grams) raising the pH to 8.43. After 24 hours, the pH of Skinny stabilized at 7.29.

Figure 4. Temporal variations in digester pH.

Post Recovery

The pH and percent total solids of the seeding digested sludge were 7.32 and 3, respectively. After the sludge addition, the pH of Chunk and Skinny dropped to 6.84 and 7.27, respectively. Over the remaining digestion time, the pH in both digesters decreased, but never got below the optimum minimum (~6.4) for anaerobic digestion (Monnet et al. 1995) (Figure 4).

Total, Volatile and Fixed Solids

Prior to recovery, the total solids content of both digesters fluctuated by less than +/- 1.2%. (Figure 5). The first data point for chunky is omitted in this observation because it was taken before the water addition. The volatile solids of Chunky and Skinny

were 86% and 95%, respectively. The amount of fixed solids in Chunky was approximately three times greater than the fixed solids in Skinny, which agrees with the substrate treatment and type.

Post recovery, the percent of total solids decreased in both digesters. Chunky dropped from 17.2% to 11.2% and Skinny dropped from 19.7% to 11.9% over the course of 30 days (Figure 6). The solids content in Chunky continued to reduce the entire post recovery time. The standard deviation for 13 of the 14 data sets was less than 1.0, meaning the sampling results were consistent. The volatile solids of Chunky and Skinny were 86% and 95%, respectively. The amount of fixed solids in Chunky was approximately three times greater than the fixed solids in Skinny, which agrees with the substrate treatment and type. The second sets of volatile solids samples were taken 30 days after the first set. The volatile solids in Chunky and Skinny decreased to 69% and 77%, respectively.

Figure 5. Temporal variations in total solids.

Biogas

Gas production was initially very slow, but accelerated once the digesters were moved from the fume hood to the window. At day 28 of the experiment (day 10 for

Skinny), the first gas volumes were estimated from the digesters. As seen in Table 1, Skinny produced more gas than Chunky despite having less time, at 3.76 gallons versus 3.31 gallons. Chunky, however, had exactly double the resistance reading from the methane sensor, at 468 versus 234.

The second collection dates are post pH control and seeding. The 10-day figure for the second collection is relative to the addition of the digested sludge and not the previous collection time. In the time between the first collection and seeding, gas production was minimal and not recorded or tested. Skinny continued to outperform Chunky, requiring a quick third gas collection after the second. The methane sensor on all second and third collections read a resistance value of 1015, assumed to be the sensor's maximum value.

Gas production continued until the end of the experiment, but no data was collected. Chunky produced a small quantity of gas likely around a gallon. Skinny produced at least twice as much, but the volume was not recorded prior to the leak and subsequent emergency container transfer. More gas was produced after the transfer, but the data was considered suspect due to the high level of contamination and interference introduced.

Table 1. Biogas production.

	Days	Gas (gallons)	Gas/day (gal/day)	Days	Gas (gallons)	Gas/day (gal/day)	Days	Gas (gallons)	Gas/day (gal/day)
Chunky	28	3.31	0.118	27	2.38	0.088	-	-	-
Skinny	10	3.76	0.376	27	4.25	0.157	2	4.16	2.078

DISCUSSION

As expected, pH control was necessary. Chunky was a mix of vegetarian food waste with the following dominant food types: leafy greens, potatoes, yams, bread, onions, coffee grounds, eggshells, banana peels and citrus peels. Citrus and coffee are acidic and probably compromised the neutrality of the digester from the beginning.

Additionally, Zhang (2010) hypothesized that the breakdown of proteins creates Ammonia, which becomes a natural buffer in an anaerobic food waste digester. Unfortunately, the substrate lacked a significant protein content that is thought to have further aggravated the already low pH. Food waste has a high content of organic soluble matter that can easily be converted into volatile fatty acids (VFAs) (Li et al., 2010). During this time, it is assumed that the contents of Chunky started a fermentation process. Demirel & Yenigün (2002) found that an ethanol-type fermentation took place at pHs lower than 4.5.

Skinny was comprised of the relatively the same types of food as Chucky, but approximately 50% of the substrate was coffee grounds. It is assumed that the coffee ground created the acidic environment (Kozuchowska & Evison, 1995). Skinny is assumed to have had the same problems as Chunky: acidic substrate, low protein content, and lactic acid domination.

As previously mentioned, an acidic environment will prevent the growth of certain bacteria species. Lactic acid is seems to be the dominant fermentation product of food waste (Zhang et al, 2010). Studies have shown that lactic acid tends to dominate the VFA concentration at all but neutral pH. Thus it is suspected that lactic acid dominated the VFA concentration in the digesters pre-recovery, which in turn prevented methane

production. The gas that was produced pre recovery was suspected to be carbon dioxide created during the organic matter breakdown.

The carbonate system test revealed that the system was not being buffered by the carbonate system. The titrations indicated that Skinny had a higher endpoint than Chunky and would require a greater amount of strong base (53 additional grams) to achieve the desired pH of 7. A day after the initial base addition, the pH of both digesters again dropped below 7. The second addition of strong base in Skinny was three times greater than Chunky (~75:25 grams). This prompted a reevaluation of the titration curves. Skinny almost appears to have two inflection points and as already noted has a higher endpoint, which could account for the amounts needed in the second addition.

The continued decrease in pH (post recovery) is attributed to the total and volatile solids content of both digesters. Solids content in the range of 20-40% is considered a “high solids content digester” by Monnet (2003). Both digesters had solids contents approximately twice the amount of the high solids digesters, thus there was still a large amount of organic solids that could be broken down. The volatile solids percentage decrease in both digesters was attributed to ongoing breakdown of organics. This decomposition continued to produce more VFAs and lowered the pH. Since the digesters were at a neutral pH, the production of VFAs were in balance with each other and the lactic acid was not dominating the system (Zhang et al, 2010).

Both digesters never appeared to reach an optimization or completion point. Besides the aforementioned reasons, it is believed that temperature was the main inhibitor to the digesters. They were operating at a room temperature and not mesophilic temperature. This alone could have prevented the bacteria growth necessary for digestion.

Many of the developing countries that currently utilize small-scale digesters are located in hot and arid areas.

In all, pH control appears to be a necessary part of food waste anaerobic digestion. The high organic content in a vegetarian food waste digest (in comparison to a wastewater digester) creates an environment that is going to be acidic from the breakdown of organics without the help of natural buffers. Future experiments should be conducted with substrates containing proteins to see if pH control could be avoided. The research of Zhang (2010) suggests that this might be possible; though obtaining that particular composition of substrate might be impractical. Temperature control could also greatly impact the digester optimization. Considering the unpredictability of most food waste streams, pH control is suggested for small-scale anaerobic digestion and methane production.

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Appendix A

Figure 6. Titration curve for Skinny, pH vs. grams of strong base added.

Figure 7. Titration curve for Chunky, pH vs. equivalence per liter of strong base added.