

Anaerobic Wastewater Treatment Final Report

Walker Grimshaw, Fanny Okaikue, Sushil Shanbhag

August 30, 2013

Abstract

The summer 2013 wastewater treatment team was the first group in AguaClara to explore wastewater treatment for developing countries. The long term goal of the wastewater treatment research is to apply the governing concepts of AguaClara: Drinking Water to the sustainable treatment of wastewater. This involves small-scale treatment strategies that utilize minimal energy and treat water of greatly varying flow. The technology must be transparent and easily operable by an individual with minimal training. The research in the summer of 2013 attempted to initially design and construct multiple upflow anaerobic sludge blanket (UASB) reactors to better understand the operation of such an anaerobic technology. Additionally, anaerobic granules were studied for their makeup and metabolic processes. Throughout the summer, two reactors were constructed, one of which was modified for use with a support media, in this case sand. During operation, COD removal and gas production were monitored, both of which initially reached a high level before declining greatly until the reactors were abandoned. Each reactor was operational for approximately one month. Future research will work to improve treatment efficiencies and maintain a constant effluent quality through use of support media and further investigation of the metabolism of anaerobic bacteria involved in wastewater treatment.

Part I

Literature Review

The literature review can be found in the accompanying document on the anaerobic wastewater treatment page.

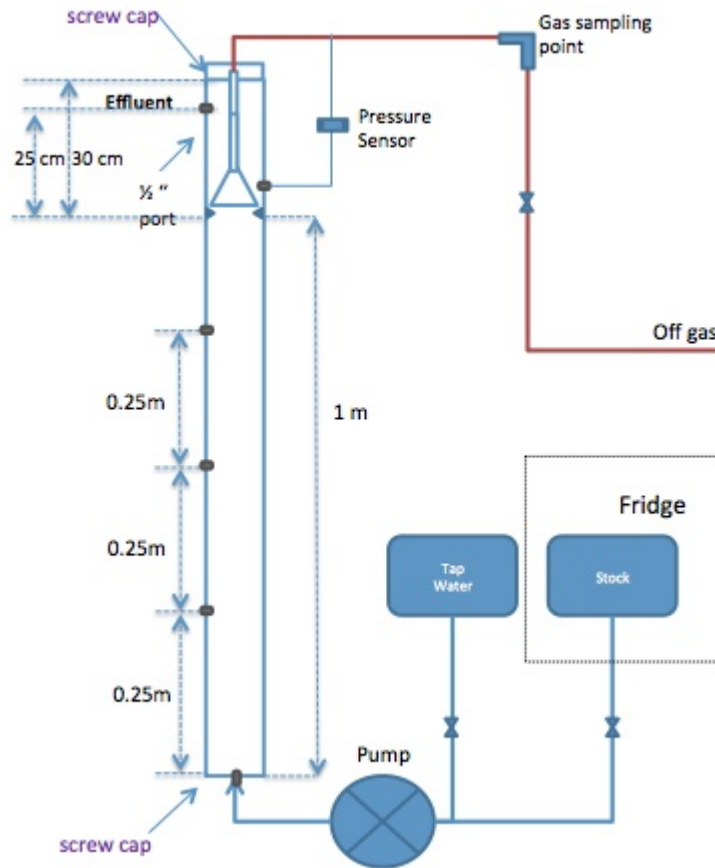


Figure 1: Design of Reactor 1.0

Part II Methods

1 Reactors and Operation

Two reactors were constructed over the summer, using 1.5 inch inner diameter clear PVC pipe. Each reactor had a one meter in length vertical portion followed by a three phase gas liquid solid (GLS) separator. The designs of each reactor and GLS separator are shown in Figure 1 and Figure 1. Initially, the reactors were operated under an upflow velocity of 0.2 mm/s and a hydraulic loading rate of 13 mL/min. This led to a hydraulic retention time of approximately 1.5 hours for the vertical portion of each reactor.

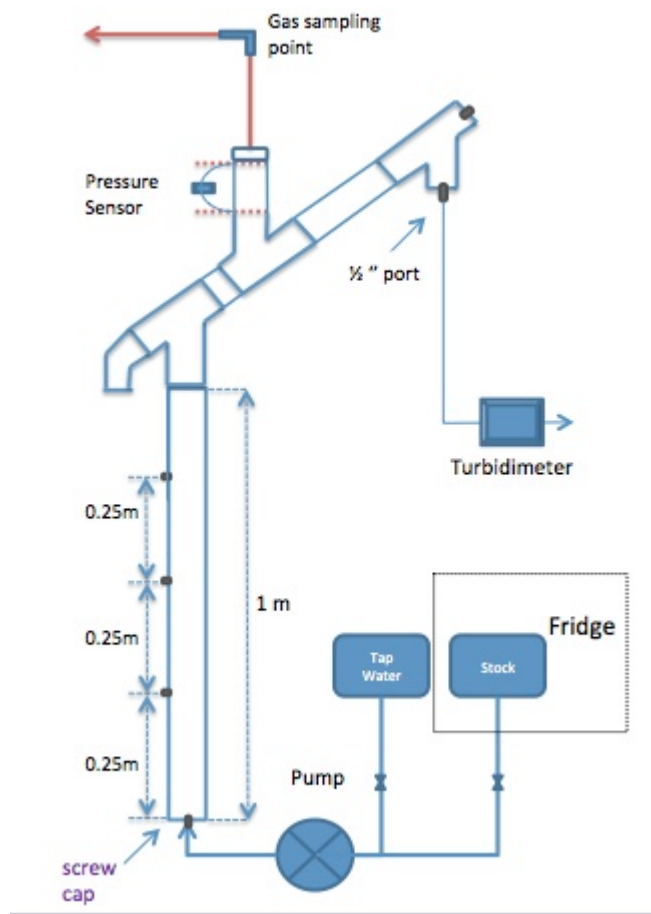


Figure 2: Design of Reactor 2.0

2 Measuring Methane Production

As shown in Figure 1 and Figure 1, each reactor was designed with a gas chamber and pressure sensor to allow for the measurement of gas production. In reactor 1.0, gas was collected within the inverted funnel while the differential pressure was monitored between the gas line above the gas chamber and a point approximately 20 cm below the effluent port. The measured differential was then the height difference between the lower pressure port and the liquid-gas interface within the gas chamber. The data was collected by process controller such that the height of the effluent port was 0 cm and height increased downward. During operation, the gas chamber remained closed until the differential height was 15 cm. At this point, an automated valve would open, releasing the gas until the height reached 5 cm, at which point the valve would then close. The height measured by the pressure sensor was logged to Microsoft Excel every 30 seconds, and this data was analyzed to determine the daily gas production for the reactor.

The gas chamber of reactor 2.0 was designed in a similar fashion to that of reactor 1.0. Figure 1 shows the vertical gas chamber extending from the tube settler. Initially, the pressure differential for this gas chamber was measured between two ports located 10 cm apart on the gas chamber, but modifications were made to replace the gas chamber cap with a removable screw cap and the differential was changed to measure between the lower port and the gas line, as in reactor 1.0. Data was gathered similarly to measure gas production.

For both reactors, the determination of the methane fraction of total biogas produced was done using a Hewlett Packard Gas Chromatograph. Once daily during operation, a 100 microliter sample was taken from the gas line of each reactor, upstream of the solenoid valve, and injected into the gas chromatograph. Elution times and peak areas were recorded. The methane partial pressure was calculated from the peak areas by creating a standard curve of known methane partial pressures. To create the standard curve, five 27 mL serum bottles were sparged with nitrogen gas then known amounts of methane were injected before gas samples were submitted to gas chromatography.

3 Synthetic Wastewater

The synthetic wastewater used in the experiments was modeled after the synthetic wastewater used by Aiyuk et al. The constituents of the wastewater and appropriate concentrations are shown in Table 3. During operation, the concentrated stock was pumped from a refrigerator for 4.5 seconds of each minute and the rest of the time tap water was pumped into the reactor to dilute the concentrated stock to 500 mg/L COD. 15 mL samples were taken from the influent and effluent of each reactor daily and used for COD measurements. COD concentration was determined spectrophotometrically through the use of a phenol solution COD test kit. As with the methane measurements, a standard curve was created to correlate UV absorption with COD concentration using

Urea	1600 mg/L
NH ₄ Cl	200 mg/L
Na-acetate	1357 mg/L
Peptone	300 mg/L
MgHPO ₄ ·3H ₂ O	500 mg/L
K ₂ HPO ₄	305 mg/L
FeSO ₄ ·7H ₂ O	100 mg/L
CaCl ₂ ·2H ₂ O	120 mg/L
Starch	2100 mg/L
Milk Powder	2000 mg/L
Yeast Extract	900 mg/L
Vegetable Oil	500 mg/L
CuCl ₂ ·2H ₂ O	10 mg/L
MnSO ₄ ·H ₂ O	2 mg/L
NiSO ₄ ·6H ₂ O	5 mg/L
ZnCl ₂	5 mg/L

Table 1: Wastewater Constituents

five samples of known COD concentration.

4 Batch Tests

Three sets of batch tests were performed to determine the theoretical gas production of the anaerobic granules when metabolizing synthetic wastewater. All tests were performed in 160 mL serum bottles with rubber septa. Tests were performed in duplicate, with each bottle receiving 50 mL wastewater and either 16.7, 25, or 40 g granules, and then sparged with nitrogen gas. In the first set of experiments, gas production was measured using a frictionless glass syringe inserted into each bottle daily. The pressurized serum bottles pushed the syringe to an equilibrium point and the volume of gas in the syringe was recorded as the gas produced that day. Gas samples were also submitted daily to gas chromatography to determine the methane partial pressure.

The second and third sets of experiments used pressure sensors and process controller to measure gas production instead of the glass syringe. The internal pressure of the serum bottles was recorded every minute by process controller. Additionally, the accuracy of each pressure sensor was measured and accounted for by injecting 10 mL of air into a closed serum bottle seven times and recording the measured pressure change as compared to the expected pressure change.

During these experiments, only few gas samples were taken due to the tendency of sampling to cause leaks in the septa. The difference between the second and third sets of batch tests was the use of tap water instead of wastewater in the final experiment to measure the endogenous gas production of the granules.

5 Granule Characterization

Because the concentration of volatile suspended solids (VSS) in the reactor can be more closely correlated to the amount of active biomass, the mass fraction of water, VSS, and FSS (fixed suspended solids) in a sample of wet granules was calculated. First, the wet granules were weighed in a ceramic crucible then placed in an oven at 105 F for 24 hours. The difference in weight before and after drying was the water weight. The solids and crucible are then submitted to a temperature of 550 for 1.5 hours, and the difference in weight is the VSS of the sample. All leftover is FSS. Additionally, granule samples were analyzed at the Cornell Nutrient Analysis Laboratory.

6 Reactor 2.1

Few modifications were made to the reactor between 2.0 and 2.1, except that the cap that had previously been glued to the gas chamber was cut away from the structure and replaced with a screw cap to allow access to the reactor for cleaning and easier inoculation. The reactor operation, however, was significantly different from reactor 2.0. The reactor was inoculated with approximately 300 g of granules and 500 mL sand. The sand had an average diameter of 0.5 mm. To fluidize the sand bed and allow biofilm growth on the sand grains, the contents of the reactor were recycled at a flow rate of approximately 740 mL/min. The normal wastewater and tap water were pumped at 6 mL/min instead of 13 mL/min to avoid washout. Due to an inability to use the normal lab space and process controller, reactor 2.1 is currently being operated with the same recycle flow, but the wastewater is only flowed through the reactor once a day at 12 mL/min until 1.5 L of double concentration wastewater has been pumped into the reactor. The gas is released from the gas chamber every day.

Part III

Results

The results described below are primarily qualitative, as the data files are currently inaccessible.

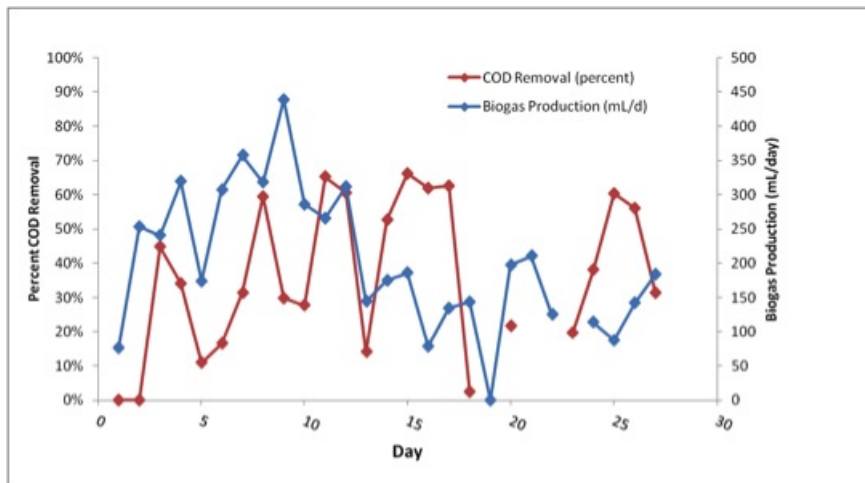


Figure 3: COD and Gas Production Results for Reactor 1.0

7 Reactor 1.0

Reactor 1.0 was operated for approximately one month before disassembly. The daily gas production and COD removal efficiencies are shown in Figure 7. It would be expected that the removal efficiency and gas production would positively correlate, but this is only evident in the collected data for the first 12 days, at which point the removal efficiency and gas production become quite erratic. The gas production, even when it did correlate well with the COD removal, was far less than expected from the amount of COD degraded by the granules. At 30 C, the theoretical methane yield from the complete degradation of COD is 395 mL/g COD, and according to this theoretical value, the gas collected in the reactor was on average 10% of the gas produced. Through qualitative monitoring of the gas production and capture in the reactor, it was apparent this amount of gas was not lost simply through leaks in the reactor. Further investigation needs to be performed to improve gas capture efficiency.

8 Reactor 2.0

The performance of reactor 2.0 was similar to that of reactor 1.0 in that gas production initially increased but only stayed at a high level for a short period of time before declining. Also, there was little correlation between gas production and COD removal, again indicating a lack of understanding of gas production in the reactor and possible gas escape in the form of leaks or dissolved methane.

Both reactors witnessed large amounts of clogging and related biomass washout. Due in part to the small diameter of the reactors, large clumps of granules would stick together and prevent the passage of gas up through the reactor. Instead,

the clumps became buoyant and rose through the reactor. In reactor 1.0, enough gas pressure would accumulate in the granule clumps to push the gas chamber out of the reactor and clog the effluent line. In reactor 2.0 the clumps would rise until reaching the tube settler, at which point the clumps broke apart and some of the biomass would return to the active part of the reactor while the rest would rise to the gas chamber or again clog the effluent line. The effluent clogging was exacerbated by the attempted use of turbidity meters. The turbidity meters used were designed for flows greater than 30 mL/min, so the flow of 13 mL/min was insufficient to continuously flow through the turbidimeter without clogging.

Additionally, after the initial period of high COD removal and gas production, the granules quickly began to disintegrate and become fluffy in nature. The fluffy granules were more buoyant than the original granular inoculum and thus more likely to washout. It is also thought the fluffy granules are less effective at metabolizing the wastewater due to the coincidence of granule disintegration and weak reactor performance.

9 Batch Tests

The measured gas production from the first set of batch experiments showed an initial high level of gas production in all six serum bottles. The two reactors with the most biomass initially produced the most gas, as would be expected, but they also continued to produce gas at a level higher than the other reactors. It was decided the use of the frictionless glass syringe to measure the daily gas production was both inaccurate and caused leaks in the septa of the serum bottles, causing even greater inaccurate measurements. The results of these batch tests have been discarded.

One day after inoculation of the second set of batch reactors, gas samples were taken from each bottle and analyzed using the gas chromatograph. This caused two of the reactors to leak and so they were restarted on day 3. Again, the reactors with the most biomass produced the most gas throughout operation and achieved total gas production levels far higher than theoretically expected from the initial COD concentration in the bottles. When the data for the batch tests was normalized to show the gas production per day per g VSS, all reactors behaved quite similarly.

The third set of batch tests was performed to determine the endogenous gas production of the anaerobic granules. As before, when normalized, all batch tests performed similarly in terms of gas production per day per g VSS. This endogenous gas production also accounted for roughly half of the gas production in the previous experiments, though the gas produced in the second set of experiments was still approximately 150% of the theoretical prediction.

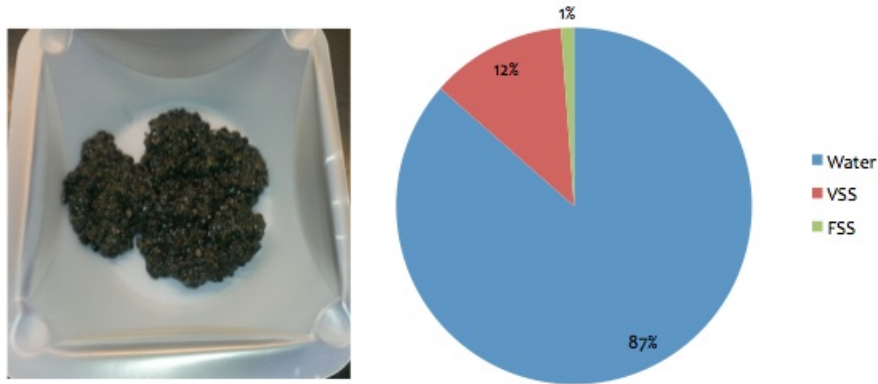


Figure 4: Granule Characteristics

10 Granule Characterization

As shown in Figure 10, wet granules are 87% water and, of the solids, over 90% are volatile. The results of the test performed at the Cornell Nutrient Analysis Laboratory show the primary constituents of the FSS within the granules are Iron, Calcium, Potassium, Sulfur, and Phosphorous.

Part IV

Future Directions

As there have been considerable problems with clogging of the reactors, future semesters will work to design and construct lab-scale reactors less susceptible to clogging failures. One manner in which this could be done would be to construct larger reactors. However, larger reactors cause greater constraints due to costs and material availability. The price of clear PVC pipe and joints increases exponentially with the diameter of the pipe and some pieces, including expansions, are not made of clear PVC. Construction of the reactor using sheets of acrylic was also considered, though constraints were met in the size of the laser cutters available at Cornell. Another attempt at preventing clogging in the reactors was implemented in reactor 1.0 in the form of recycling flow in a small section of the reactor. Flow was recycled between ports 2 and 3 on the reactor and served to agitate the clumps that had previously arisen. Though the large plugs of granules no longer reached the GLS separator with large amounts of gas trapped within, groups of sticky granules did still rise into the gas chamber and disrupt the gas collection and measurement process. A current failure to solve the issues of clogging and biomass loss with the use of granules has led to further exploration of using support media in the reactor. It is hypothesized

that biofilm growth on support media can be more easily controlled than the development of granules and that washout can be more easily prevented with support materials due to the comparatively low density of granules.

Reactor 2.1 is the first reactor to use any support material. It would be beneficial to build similar reactors and test the use of different support materials, both fluidized and non fluidized. Though fluidization ensures good contact between the biomass and substrate, this often requires recycling and thus an energy input. Future work should strive to decrease energy inputs into the reactor so as to be most applicable in developing communities. Possible manners to achieve little recycle would be to use a support material for which fluidization would require little or no extra energy as well as increasing the height of the reactor so a high upflow velocity will still allow for a sufficient hydraulic retention time (HRT). It will be beneficial to construct models of the fluid dynamics within the reactor, especially during biofilm development. The models will serve as a basis for estimating the appropriate amount of support material to use in the reactor and the flow rate needed to fluidize the bed.

In the future, the anaerobic metabolism of the wastewater will also be further explored to improve the treatment efficiency within the reactors. Currently, the reactors are capturing roughly 10% of the theoretically produced methane. Though this corresponds with data reported in other UASBs and AFBRs, this represents a part of the anaerobic treatment process which this research can greatly expand. One of the first steps in this process will be to measure the volatile fatty acids (VFAs) in the reactor. This will illuminate if the substrate is only being partially degraded during treatment. Determining what part of the treatment process is the rate limiting step, mass transfer for instance, is integral to improving upon current anaerobic treatments for the developing world.