

Expanded Granular Sludge Bed (EGSB) Team Report, Spring 2016

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Abstract

The Expanded Granular Sludge Bed (EGSB) team was created to work within the wastewater subteam to design and run new, bench-scale, high rate anaerobic reactors. New reactors were designed to create a system with increased upflow velocity of influent, a fluidized bed, and decreased hydraulic retention time without decreased granular retention. Reactors were designed with simple operation in mind, with narrow modules in series rather than a single large reactor with recycle. The reactors were inoculated following abiotic testing of pumping rates, connection seals, and methane sensors. Immediately after inoculation, the granules began to form blockages and back up the reactor. Various forms of agitation seem to alleviate the problem, and automated solutions to the blockage problem has been proposed. In addition to blockages, the first module of the reactor was acidifying due to the low hydraulic residence time and relatively high specific organic loading rate. However, the following three modules were observed producing significant amounts of methane via the sensors, and at the end of an uninterrupted week of operation a COD test indicated about 40 percent total COD removal. With improved methane sensor calibrations and a blockage prevention system, the bench-scale setup for high rate anaerobic treatment could potentially become a very versatile tool for testing the limits of anaerobic wastewater treatment and methane bioenergy reclamation.

Introduction

AquaClara's sustainable wastewater treatment teams work to develop low-energy, low-cost, and compact systems. The Expanded Granular Sludge Blanket (EGSB) team was developed this semester to build on and modify previous Upflow Anaerobic Sludge Blanket (UASB) team work involving chemical oxygen demand (COD) removal and methane production and capture. The EGSB team was created to address the desire to decrease hydraulic retention time (HRT), increase solid retention time (SRT), and increase sludge bed fluidization to more effectively use reactor space. EGSB reactors use upflow velocities that can be as much as ten times faster than those in UASBs (Liu et al., 2007). The slow velocities in UASBs lead to the creation of a very dense sludge bed that remains at the base of the reactors, and long HRT times are required to allow for adequate periods of contact between granules and wastewater. Higher upflow velocities in EGSBs allow the bed to fluidize and fill more space within the

reactor, increasing the time of contact between sludge granules and wastewater (Seghezzo et al., 1998). Higher upflow velocities and the resulting lower HRTs in EGSBs can be more cost-effective because they allow for smaller reactors and lower capital costs.

Unfortunately, there are a number of limitations on anaerobic waste treatment that have hindered the use of EGSBs over UASBs. While EGSBs have higher theoretical efficiency, in practice they are very sensitive to changes in the wastewater strength (Fang et al., 2011). This is because of the low growth rate of the anaerobic bacteria present in the reactors. In order for anaerobic reactors to reliably treat waste, they must contain a high concentration of bacteria (Jewell, 1987). This high concentration ensures that even during periods of slow growth due to low organic loading, the reactor meets the desired treatment standard. The concentration of granules in UASBs is consistently high as the granules sit in a large bed, however in EGSBs the granules are either fluidized or attached to a filter media. While such an arrangement allows a shorter HRT, it risks microbe loss in the effluent. Such losses hurt the treatment efficiency of EGSBs, as the regrowth of microbes in the reactor is slow (Jewell, 1987). The current solution to this problem has been to use solids capture at the end of the reactor to ensure a high concentration of microbes in the reactor at all times. The EGSB team aims to apply AguaClara's plate settlers as solids capture for a simple EGSB, which could be easily fabricated and employed at low cost in high temperature regions.

Literature Review

Upflow Anaerobic Sludge Blanket Reactors (UASBs), are increasingly being investigated as a cost-effective way to treat organic wastes (Jewell et al., 1981). This is due to the combined advantages of anaerobic digestion and upflow reactor configurations, which allow for the rapid treatment of wastes with minimal sludge accumulation within the reactor. This is advantageous to conventional activated sludge sewage treatment, as it eliminates the costly processes of aeration and sludge disposal (Jewell, 1987). Despite these substantial advantages, upflow anaerobic sludge blankets still face a few major obstacles limiting their widespread application to municipal sewage treatment. First, anaerobic reactors are temperature dependent, and suffer from low reaction rates at low temperatures. For temperatures as low as 10 degrees Celsius this limitation can be overcome with high cell retention time, but such measures often involve high cost membranes or recycle lines (Lee et al., 2015). Second, the upflow velocity as well as HRT of UASBs are currently limited by the solids retention time required for satisfactory removal of biochemical oxygen demand (BOD) from sewage. The solids retention time is process limiting because the kinetic advantage of upflow reactors does not extend to suspended BOD. This is because such particles of BOD require solubilization via hydrolysis before they can be degraded, and the more hydrolysis required the more reactor performance suffers. As a result, high upflow reactors are conventionally considered an option only for dissolved BOD streams rather than suspended BOD streams.

The first short-coming of UASBs should not pose a large issue to AguaClara's current activities, as tropical climates such as Honduras are naturally well-suited for high rate anaerobic digestors (Seghezzo et al., 1998). However, the issue of

solids retention in UASBs is a non-trivial problem, as issues in this area can cause reactor unreliability. Conventional UASBs utilize a large retention time, in order to maximize solids capture and with it the variable response capabilities of the reactor. A typical superficial upflow velocity recorded in a bench scale experiment from 1990 was 0.5 m/h (0.14 mm/s) (Kosaric et al., 1990). This velocity is nearly ten times lower than that of an AguaClara settling tank, and resulted in a hydraulic residence time of 2 hours (in their experiment). However, this results in a considerable portion of dead space in the upper portion of the reactor used strictly for settling. In order to better utilize this space, a modified UASB with an expanded bed of fluidized granules was developed in order to maximize active reactor surface area.

A recent study using palm oil was done comparing the performance of the conventional UASB to that of the modified Expanded Granular Sludge Bed (EGSB), and the results found that the EGSB performed nearly as well as a conventional UASB with considerably lower HRT (Fang et al., 2011). The upflow velocities at which anaerobic granules typically fluidize have been reported (Liu et al., 2007) to be in the range of $4\text{-}8 \text{ m/h}$ (or $1.1\text{-}2.2 \text{ mm/s}$ for comparison with AguaClara sedimentation tanks). This would result in an HRT between 7.5-15 minutes on the same scale as the 1990 bench scale upflow anaerobic reactor (Kosaric et al., 1990). EGSBs appear to be a natural evolution of the UASB in all areas, except for a notable failure to effectively treat suspended BOD. Due to the short HRT of EGSBs, suspended wastes pass through the reactor more rapidly than they can be hydrolyzed by the suspended granules (Seghezzo et al., 1998). This obstacle is one that the current team hopes to overcome using AguaClara's knowledge of plate settlers by maximizing the exposure time of soluble waste to the expanded sludge bed such that hydrolysis is no longer limiting.

Previous Work

The AguaClara Wastewater Group began in the summer of 2013 with the goal to research sustainable wastewater treatment technology. In the Fall of 2013, six reactors were constructed. Three of the six reactors were UASBs and the other three were Anaerobic Fluidized Bed Reactors (AFBRs). The original reactors were 1 m tall, 1.5 in diameter vertical columns with tube settlers and biogas collectors on the arm of the tube settler. The reactors were fed with 500 mg/L COD wastewater at a rate of 5 mL/min , thus leading to a HRT of approximately 3.8 hr . The upflow velocity was 0.07 mm/s . The Fall 2013 group began operation of these reactors, but was unable to collect a significant amount of gas production data due to leaks in the reactors and the lengthy startup time required for steady state operation. For the AFBRs, the group developed mathematical models for particle fluidization and settling within the reactor. The group proposed a new gas chamber sealing method based on the coupling of a pressure sensor with Process Controller that would potentially only release accumulated biogas once a certain gas pressure had been reached. Finally, the Fall 2013 group used confocal microscopy and chemical staining in an attempt to characterize the granules within the reactor. The group was able to identify regions within the granule involved in active DNA and RNA synthesis as well as groups of aggregated methanogens.

The Spring 2014 group split into three subgroups: a UASB operation improvement group, a gas production and collection improvement through design and scaling modification group, and an aerobic treatment options group. The same reactors were run at half flow rate and double influent COD concentration. Thus the HRT was extended to 7.6 hr. Gas chromatography was used to monitor the amount of methane produced by the reactors throughout the semester. The Spring 2014 group faced several problems with respect to reactor performance: inconsistencies between theoretical and experimental gas production, inconsistent COD feed concentration delivery, and vessel leakage. These issues were provided as explanations as to why the experimental data did not match theoretical predictions for biogas production. In an attempt to fix the air tightness issue, the group designed two tests to identify leaks in the reactors, and tried to repair leaks with Teflon tape and covering joints with parafilm, which was unsuccessful. The group eventually sealed the connections with epoxy for reactors 2.4 and 2.5. Reactor 2.4 remained airtight throughout the semester, but reactor 2.5 began to leak a few weeks into operation. Effluent testing showed that methane was also lost in the form of dissolved methane leaving the reactor in the liquid phase.

The Fall 2015 group split into two subgroups: GSBR team and UASB team. The UASB team mainly focused on the sealing of the reactor. Two of the previous UASB reactors (2.5 and 2.3) were cleaned and run at a flow rate of 6.25 mL/min. The HRT was approximately 3 hours. The team designed a series of tests to find the leakage and calculated the biogas loss rate to be 3.94%. The biogas production rate and COD removal rate were measured and compared with previous tests. The fraction amount of methane in biogas was measured to be 48 to 59% of theoretical methane production. Tables 1 and 2 include data from Fall 2015 to quantify methane production and COD removal from reactor 2.5.

	Fall 2013 Rctr 2.4	Fall 2013 Rctr 2.6	Spring 2014 Rctr 2.4	Fall 2015 Rctr 2.5
Methane Prod. (mL/day)	116.8	139.5	227.5	141.8

Table 1: Methane Production Fall 2013 to Fall 2015

Date	Total Gas Production (mL)	Volume Fraction Biogas	Volume Methane Produced (mL)	Influent COD (mg/L)	Effluent COD (mg/L)	% COD Treated	% Theoretical Methane Production
11/29/2015	218.1	~65	147.5	789	129	84%	59%
11/30/2015	149.4	~65	97.1	632	72.9	88%	48%
12/1/2015	142.9	65.5	93.3	2360	109	95%	12%

Table 2: % COD treated and % Theoretical Methane Production

The Spring 2016 group split into three subgroups: GSBR, UASB and EGSB team. The EGSB group is redesigning the reactors. Major problems with Fall 2015 UASB reactors included the lack of air-tightness, along with subsequent loss of biogas, and long HRTs of around 7 hours. These problems compelled the design of secure biogas capture chambers, and construction of smaller reactors in series to have lower HRT and increased bed fluidization.

Methods and Discussion

1 Pretests

A pretest apparatus was made to run a granule fluidization test and terminal velocity test. The goal of the pretest was to determine a design flow rate for the reactor and a design granule terminal velocity for the tube settler. According to the results, the minimum fluidization flow rate was found to be between 40 to 60 mL/min in 1 *in* diameter pipe. The terminal velocity varies for different size of the granule. The minimum terminal velocity, measured from several very small granules, was approximately 0.3 mm/s . However, the design terminal velocity was set at 0.1 mm/s to include a factor of safety and minimize granule washout. In addition, the dimensions of the granules were measured with grid paper. Granule size characterization was done so that future teams know the granules that the Spring 2016 semester worked with for fluidization tests and inoculation. Different granule size would impact the minimum fluidization flow rates and terminal velocities chosen, and future teams can compare their granules to this semester's.

1.1 Experimental Apparatus

A 1.2 *m* long, 1 *in* diameter clear PVC tube was clamped vertically. A 3.5 *cm* long cone was fabricated at the influent point to create a jet at the bottom of the column to better suspend the solids. A ruler was attached to the column for easy measurement. 100 mL of sludge was poured into the column, forming a original sludge height of 19.2 *cm*, including 1/3 of the length of the cone.



Figure 1: Pretest Experimental Apparatus

1.2 Procedure

The minimum bed fluidization rate was determined by pumping tap water up through the column with a series of flow rates ranging from 6 mL/min to 320 mL/min with a 20 mL/min interval. For each flow rate, the height of the fluidized bed was measured with ruler. As the flow rate increased, the granules became very spread out and did not create a defined bed top. Bed height was defined to be the highest point which had granules that the team did not want to have wash out.

Next, the settling velocity of a number of granules of various size were measured with ruler and stopwatch. Tap water was pumped through the apparatus in 240 mL/min to lift the granules to the top of the apparatus. When the bed was fully expanded, the pump was stopped and the granules started to fall. Granules of different size were sampled from upper half of the column, one at a time, and were measured after reaching steady falling velocity. Two people worked together to measure the settling velocity of the sampled granule, one with ruler and the other with stopwatch. The large granules which stayed in bed even when fluidized were not measured because they interact with each other when settling.

Then, 1 mm by 1 mm grid paper was used to give a rough measurement of the dimensions of the granules. A sample of about 40 granules was poured into a clear Petri dish, and 20 granules of assorted shape and size were randomly measured for diameter size. To measure diameter size, the granules were approximated to be circular. (See Figure 2)

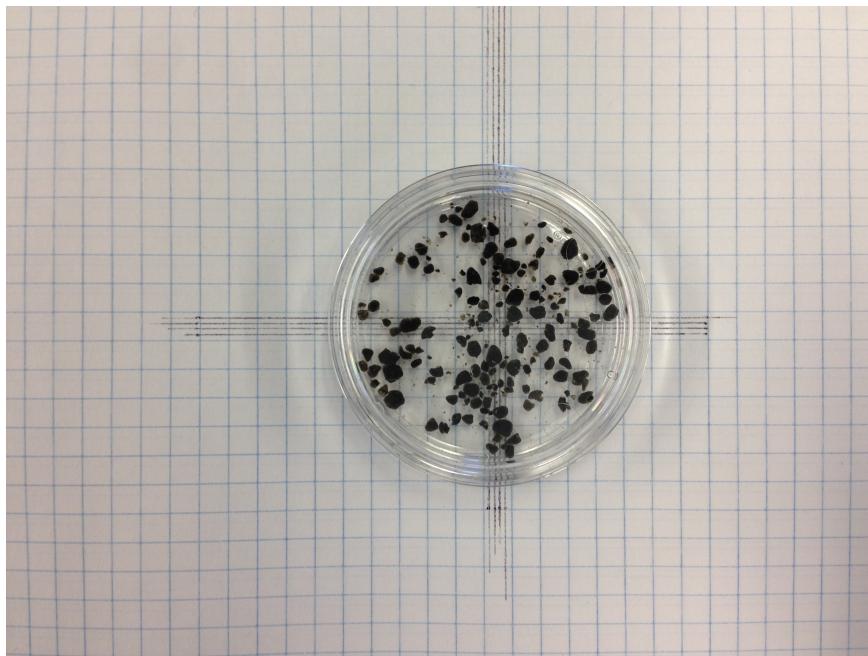


Figure 2: Granule dimensions

1.3 Results and Analysis

Table 3 shows the flow rates tested and corresponding upflow velocity, HRT, solid bed height expansion, and percent expansion. This fluidization test provided a preliminary target pump flow rate of 60 mL/min and an minimum upflow velocity at 1.79 mm/s. These numbers were chosen because at these standards, the entire bed of granules began oscillating. The bed was arranged by size, with the smallest granules at the top. At higher flow rates, the bed expanded more, but to limit granule washout that occurred at higher rates, 60 mL/min was chosen.

Flow rate (mL/min)	Upflow Velocity (Q/A)(mm/s)	HRT (V/Q)(min)	Solid Height (mm)	Percent expansion (%)
6	0.18	111.9	21	9.6
20	0.60	33.6	22.2	15.8
40	1.19	16.8	24.5	27.8
60	1.79	11.2	25	30.4
80	2.39	8.4	27	40.9
100	2.98	6.7	29.5	53.9
120	3.58	5.6	32.5	69.6
140	4.18	4.8	37	93.0
160	4.77	4.2	40	108.7
180	5.37	3.7	43.5	127.0
200	5.97	3.4	70	265.2

Table 3: Fluidization Test Result

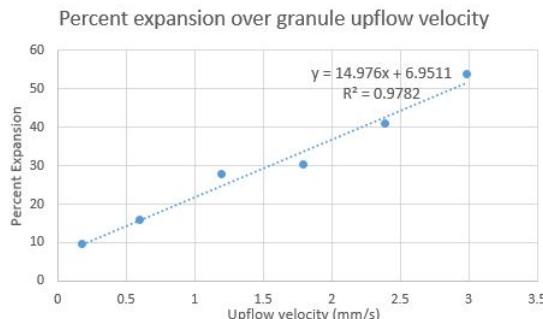


Figure 3: The Relationship Between Bed Expansion and Upflow Velocity

An upflow velocity of 1.79 mm/s is significantly higher than upflow velocities used by UASBs. Fall 2015 UASB team's upflow velocity was about 0.0912 mm/s. It was expected that velocities would need to be much higher than UASB's to allow bed expansion, and this 1.79 mm/s velocity aligns with Liu et al. work that found fluidization in the 1.1-2.2 mm/s range.

Table 4 shows settling velocities of different granule size. Measuring settling velocity of large granules proved difficult because they perform hindered settling mode in a relatively dense portion of the bed. The settling velocity of small and medium granules are easier to measure because they can be lifted up to the top of the reactor and fall down in free settling mode. Their settling velocities are within the range of 0.321 mm/s to 1.425 m/s

Granule size	Small granules			Medium granules		Large granules	
Settling velocities (mm/s)	0.666	0.321	0.476	0.395	0.871	1.425	1.299

Table 4: Settling Velocity Test Result

According to the results, 0.321 mm/s was the minimum settling velocity among all granules sampled. Because granule retention is important, an added factor of safety was then added to ensure that no desired granules get washed out, and the final working settling velocity chosen for reactor design is 0.1 mm/s .

Granule measurement showed that the average granule diameter was about 2 mm . This granule measurement will change once reactors are inoculated because the granules will grow and die, but this measurement was helpful for establishing starting parameters.

2 Reactor Design and Operational Characteristics

Results from the pretests provided values to be used in determining the reactor dimensions necessary to meet HRT and SRT goals. The reactor is comprised of 4 smaller reactors in series. Each small reactor is constructed with a 1 in pipe, connected to influent with a cone port. Water flows from the first reactor to all the others, and biogas created in each reactor is captured and measured separately. A weir is designed at the exit of the last reactor to prevent draining. Two different biogas collection and methane measurements were designed so that if the first option of using methane sensors does not work, the reactor can easily be converted to use off-gases similar to UASB's design from Fall 2015. The following schematics detail reactor dimensions.

2.1 Goals and Constraints

The goal of design is to build a reactor with sufficient HRT and fluidizes the bed effectively. The volume of the reactor is constrained by the height of the ceiling and space. Based on the HRT and upflow velocity data in table 3, the minimum upflow velocity is chosen to maximize HRT, which is approximately 1 hr with four reactors in series. In order to fluidize the bed effectively, cones 4.2 cm in length were fabricated and installed in the inlet. The angle of the tube settlers was set to be 45 degrees because of the availability of elbows and wye joints. Next, the number of reactors in series, the height of reactor and the length of tube settlers were calculated with the equations below. The gas collector is connected to the tube settler with 1 in wye joint.

Variables:

$$V_{Up} := 1.79 \frac{\text{mm}}{\text{s}} \quad N_{column} := 4 \quad HRT_{reactor} := 1\text{hr} \quad D_{reactor} := 1.05\text{in}$$

$$V_t := 0.1 \frac{\text{mm}}{\text{s}} \quad \alpha_{TubeSettler} := \frac{\pi}{4}$$

$$A_{column}(D_{reactor}) := \frac{D_{reactor}^2}{4} \cdot \pi$$

$$Q_{reactor}(V_{Up}, D_{reactor}) := V_{Up} \cdot A_{column}(D_{reactor})$$

$$Q_{reactor}(V_{Up}, D_{reactor}) = 59.998 \frac{\text{mL}}{\text{min}}$$

$$V_{reactor}(N_{column}, HRT_{reactor}, D_{reactor}, V_{Up}) := HRT_{reactor} \cdot Q_{reactor}(V_{Up}, D_{reactor})$$

$$V_{reactor}(N_{column}, HRT_{reactor}, D_{reactor}, V_{Up}) = 3.6\text{L}$$

$$H_{column}(N_{column}, HRT_{reactor}, D_{reactor}) := \frac{V_{reactor}(N_{column}, HRT_{reactor}, D_{reactor}, V_{Up})}{A_{column}(D_{reactor}) \cdot N_{column}}$$

$$H_{column}(N_{column}, HRT_{reactor}, D_{reactor}) = 1.611\text{m}$$

$$L_{TubeSettlerEST}(V_{Up}, V_t, \alpha_{TubeSettler}, Q_{reactor}, D_{reactor}) := \frac{D_{reactor} \cdot (V_{Up} \cdot \sin(\alpha_{TubeSettler}) - V_t)}{V_t \cdot \sin(\alpha_{TubeSettler}) \cdot \cos(\alpha_{TubeSettler})}$$

$$L_{TubeSettlerEST}(V_{Up}, V_t, \alpha_{TubeSettler}, Q_{reactor}, D_{reactor}) = 0.622\text{m}$$

where $V.Up$ is upflow velocity, $N.column$ is the number of reactors in series, $HRT.react$ is the hydraulic retention time for all reactors, but not including tube settlers and gas reactors. $D.react$ is the diameter of the reactors. $V.t$ is the assumed least terminal velocity of the granules, $\alpha.TubeSettler$ is the angle of incline of the tube settlers.

2.2 Reactor Design Iteration 1

The first design included methane sensors (see Figure 4). It was the first time the team used methane sensors within AguaClara. Biogas collected in the headunit of the reactor was immediately released through microbore tubing, which was then connected to a larger tube that has a constant rate of air pumped through it. The diluted methane then passed through the methane sensor and recorded a continuous set of data for methane concentration. Because this method created a free water surface in gas collector, the height of gas collector was designed to be 10 cm higher than the exit of the reactor. Also, a weir was designed at the exit of the last reactor to keep the water level in all reactors at that point. Thus the total height of a reactor was set at approximately 2.3 m.

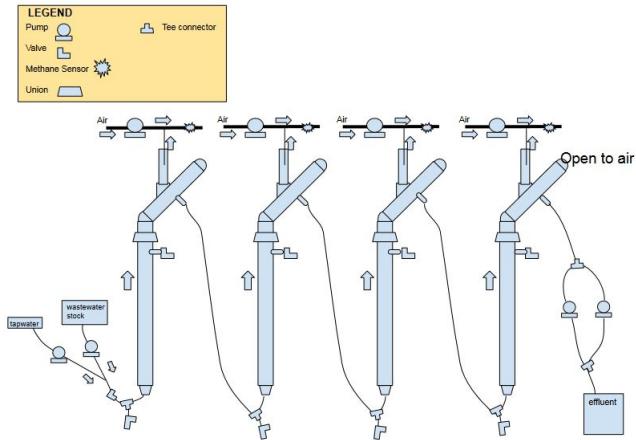


Figure 4: Reactor Design with Methane Sensors.

The second design allows biogas to accumulate in the headunit and thus push the water level down in methane collector (see Figure 5). This design is a modification of the first, and can be used if the methane sensor method does not prove viable. ProCoDA is used to program offgas events after the pressure increases to a certain point. The offgas occurs through the opening of a solenoid valve, and the offgas ends when the pressure falls below a certain point and the solenoid valve closes. Biogas production is calculated by integrating over offgas events.

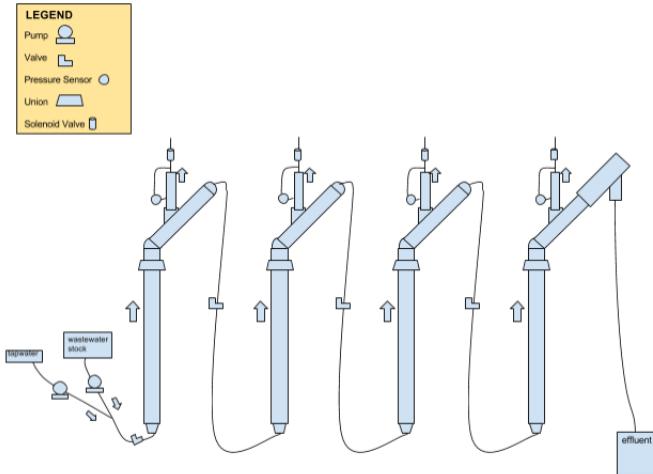


Figure 5: Reactor Design with Off-gas option.

Reactors shown in Figures 6 and 7 were constructed using these initial designs to begin abiotic testing.



Figure 6: Reactors in Series: Iteration 1



Figure 7: Reactor Side View: Iteration 1

2.3 Reactor Design Iteration 2

Abiotic testing of the reactors exposed two different issues with the design that needed to be addressed: reactor filling problems and reactor operation problems.

2.3.1 Reactor Filling

Problems with reactor filling occurred when water was pumped into the first reactor and allowed to begin filling the second one. At first, the reactors were allowed to fill in series by attaching the water line, which has very high water flow, to the first reactor. It was observed that the first reactor filled to the height of the effluent exit, leaving unfilled space in the top of the biogas collection unit. However, after half of the second reactor began to fill, water began to spill out of the uncapped top of the first reactor. The water would not drain from the first to the second even when the line was closed. It was observed that bubbles got trapped in the 1/4" tubing between reactors, preventing water from flowing down. When water was prevented from moving through the tubing due to bubble blockage, it overflowed through the open biogas head. To prevent overflow, the open biogas head needed to be covered. When covered, pressure was allowed to increase and helped push water through to the next reactor. However, this filling method required three reactors to be capped to fill all four reactors, which was inconvenient.

The first solution the team came up with was adding a t-joint at the top of the reactor exit (figure 8). The t-joints at the effluent supported decreasing lengths of top tubing from reactor 1 to reactor 4, to account for decreasing water levels due to headloss. Thus, air bubbles in line could be released and water would go through more easily. It also provided an open surface below biogas head. So if water overflowed, it overflowed through t-joint instead of biogas head. However, the t -joint did not change anything, as air still got trapped in tubing and water overflowed as before. Continued overflow made it clear that the diameter of the tubing connecting reactors was too small to allow an open channel supercritical flow in it.

The second solution was to change the method of filling the reactors. The reactors were tested by filling them all separately from the bottom, to then be connected back together for operation. When the reactors were being filled individually, the water level in tubing increased with it and air was released from the t-joint exit with no problems. However, in case any air bubbles get into tubing and cause problem in operation, the team decided to change all tubing to 3/8", to see if it is large enough to conduct open channel supercritical flow and did not trap air bubbles. The team kept the t-joints in the reactor design to prevent overflow during operation which was of major concern when working with methane sensors because they cannot get wet.

Later, the team changed all 1/4" tubing used in reactors to 3/8". The test of 3/8" tubing showed it was still not large enough to conduct supercritical flow. So the reactors have to be filled from the bottom separately afterwards.



Figure 8: T-joints added at effluent to promote escape of bubbles in tubing.

2.3.2 Reactor Operation

After filling the reactors, the team operated them under the flow rate of 60 mL/min and measured the headloss between reactors. Initial headloss calculations showed headloss would be less than 1 mm (shown in the equations below). However, once the reactors were filled and water levels in head space steadily difference in height between consecutive reactors was measured at 4 cm , as shown in Figure 9.



Figure 9: Headloss at steady operation and $1/4"$ tubing

The theoretical calculations for headloss with $1/4"$ tubing, shown below, differed greatly from observable headloss. More testing must be done to better understand the difference between calculated headloss level of 0.118 mm and observed headloss level of 4cm .

Major head loss per reactor:

Assume 1/4 in pipe diameter and 2.3 m pipe length between reactors

$$h_{f,reactor}(Q_{reactor}, D_{reactor}, L_{TubeSettler}, H_{column}) := \frac{32 \cdot \nu_{water} \cdot (L_{TubeSettler} + H_{column}) \cdot v_{Up}(Q_{reactor}, D_{reactor})}{g \cdot D_{reactor}}^2$$

$$h_{f,reactor}(Q_{reactor}, D_{reactor}, L_{TubeSettler}, H_{column}) = 0.039 \text{-mm}$$

$$h_{f,pipe}(Q_{reactor}, D_{pipe}, L_{pipe}) := \frac{128 \cdot \nu_{water} \cdot L_{pipe} \cdot Q_{reactor}}{\pi \cdot g \cdot D_{reactor}}^4$$

$$h_{f,pipe}(Q_{reactor}, ID\left(\frac{1}{4} \text{in}, 2\right), 2.3 \text{m}) = 0.038 \text{-mm}$$

Minor head loss per reactor:

Entrance (gradual enlargement):

$$K_1 := 3.1 \quad ID\left(\frac{1}{4} \text{in}, 2\right) = 0.364 \text{-in}$$

$$h_{e,1} := K_1 \cdot \frac{(Q_{reactor})^2}{\left[\frac{\pi \cdot (ID\left(\frac{1}{4} \text{in}, 2\right))^2}{4} \right]^2 \cdot g} = 0.281 \text{-mm}$$

30 degree bend:

$$K_2 := 0.18$$

$$h_{e,2} := K_2 \cdot \frac{v_{Up}(Q_{reactor}, D_{reactor})^2}{2g} = 1.176 \times 10^{-4} \text{-mm}$$

Exit :

$$K_3 := 0.5 \cdot \left(1 - \frac{ID\left(\frac{1}{4} \text{in}, 2\right)^2}{ID(1 \text{in}, 2)^2} \right) = 0.44$$

$$h_{e,3} := K_3 \cdot \frac{(Q_{reactor})^2}{\left[\frac{\pi \cdot (ID\left(\frac{1}{4} \text{in}, 2\right))^2}{4} \right]^2 \cdot g} = 0.04 \text{-mm}$$

Total Head loss:

$$h_{L,reactor} := h_{e,1} + h_{e,2} + h_{e,3} + h_{f,pipe}(Q_{reactor}, ID\left(\frac{1}{4} \text{in}, 2\right), 2.3 \text{m}) + h_{f,reactor}(Q_{reactor}, D_{reactor}, L_{TubeSettler}, H_{column}) = 0.397 \text{-mm}$$

However, after changing the tubing size to 3/8", the headloss between reactors reduced from 4 cm to approximately 0 cm in steady flow rate of 60 mL/min. It effectively reduced the water levels in the gas collectors. Next, the team decided to inoculate.

2.4 Reactor Design Iteration 3

In this iteration, the methane sensors were added to the reactors and inoculation was started. When the granules were loaded into the reactors, it altered the hydraulics in the system and caused problems. The reactor design is then further modified to solve the problems in inoculation process. The modified reactor design is shown in Figure 10.

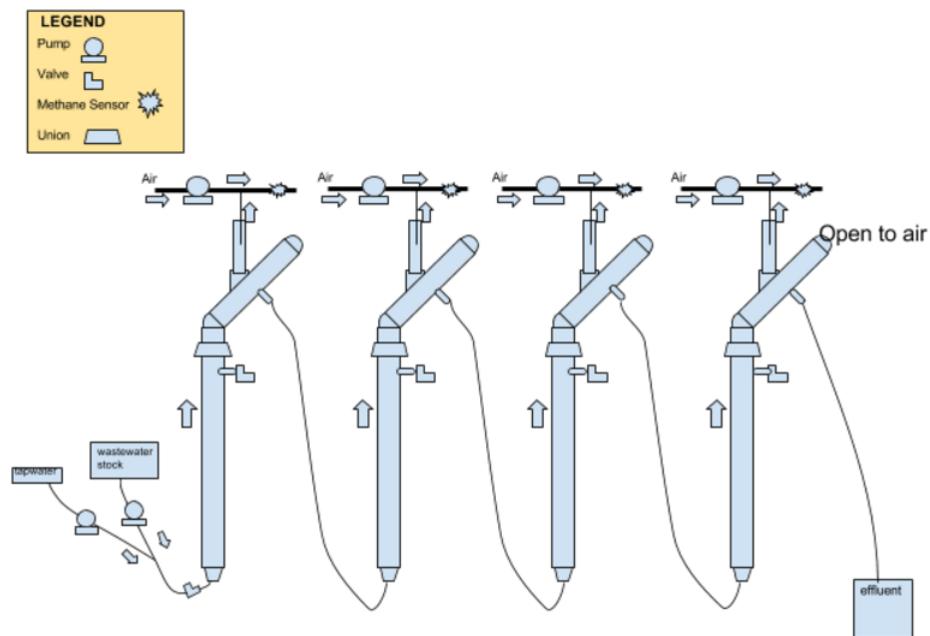


Figure 10: Reactor Design with Lower Exit and Valves under Unions

2.4.1 Methane Sensors

Biogas produced from the reactors must be analyzed for amount of methane produced. As mentioned above, previous teams analyzed methane production by allowing offgas events from the headunit, and then testing for methane using gas chromatography. A new method of measuring methane production directly using methane sensors was explored by the team in an attempt to streamline the methane sensing process.

The team purchased 4 MQ-4 model methane sensors from Zhengzhou Winsen Electronics Technology Co., Ltd. (Winsen, 2015) One methane sensor was wired to create a prototype for testing and calibration curves were created and are as shown in Figure 11.

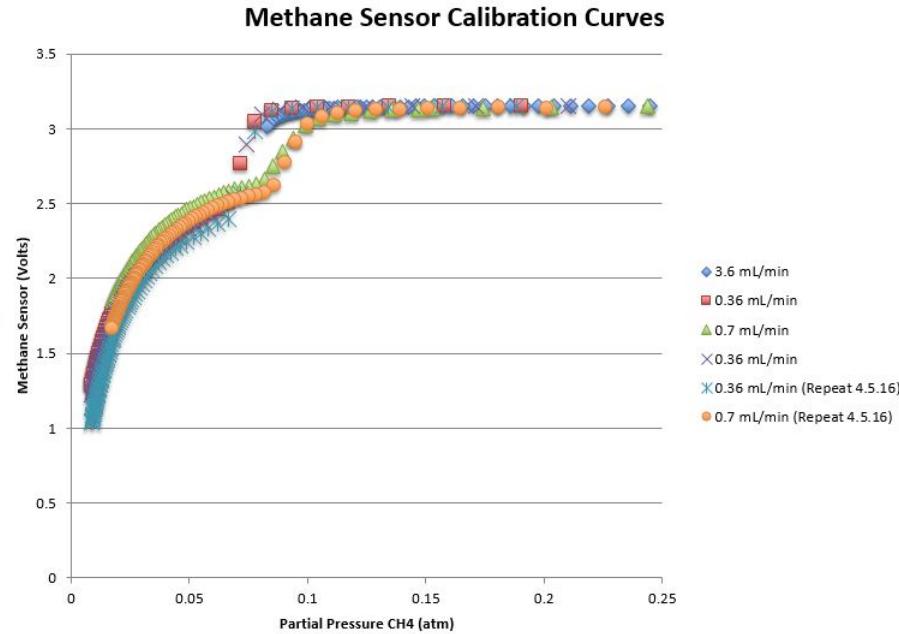


Figure 11: Methane Sensor Calibration Curve created with methane sensor prototype.

The calibration curves suggested that there was little variation in voltage reading between different flow rates within the range of 0.36 mL/min to 3.6 mL/min . The methane sensors are rated to detect gas concentrations from 200 to 10,000 ppm. A partial pressure of methane at 0.07 atm and a safety factor of 2 suggests that the methane partial pressure to be used should be 0.035 atm . If methane is assumed to be 65.5%, and each reactor produces 50 mL/day , then the expected methane production per reactor dilution demand is 0.627 mL/min .

The calibration curve can be fitted to the function below:

$$P := 0.0017 \cdot \exp(1.3739 \cdot x) \cdot \text{atm} \quad R^2 := 0.9461$$

$$Q_{\text{CH}_4}(Q_{\text{air}}) := P \cdot Q_{\text{air}}$$

in which P is the partial pressure of methane in atm, x is the sensor output in voltage, Q_{air} is the air flow rate, and Q_{CH_4} is the methane flow rate.

A methane measurement apparatus was set up to allow for dilution of the biogas coming from the reactors. A variable rpm three stop pump was set up and connected to the microbore biogas line with a t-joint before being connected to a methane sensor housing unit. The tabletop set up is highlighted in the Figure 12 and Figure 13.

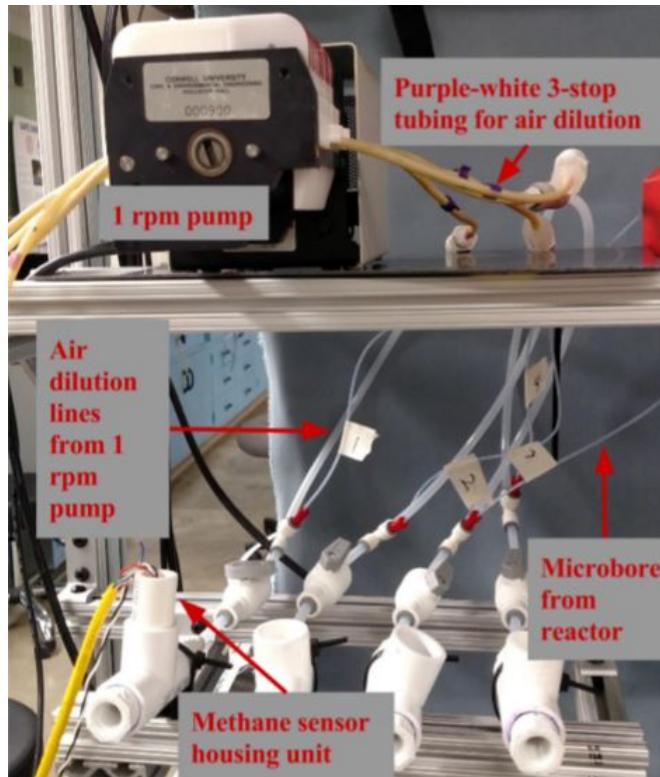


Figure 12: Tabletop set up of methane dilution system with methane sensor housing units.

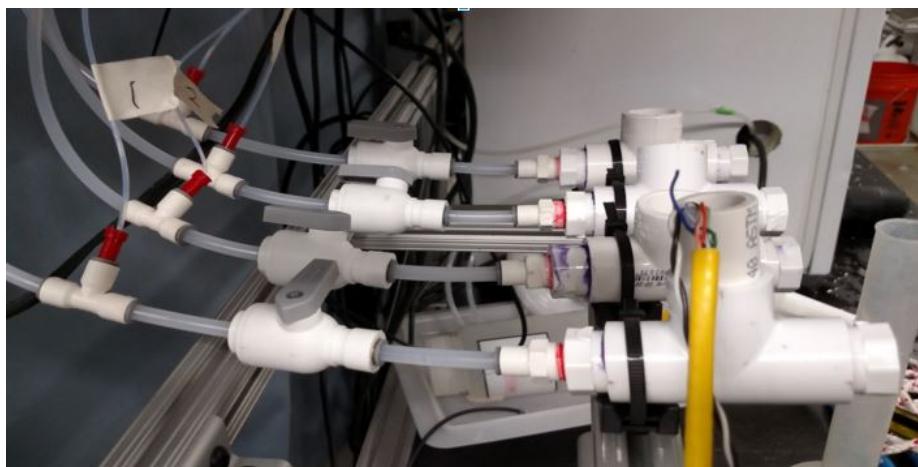


Figure 13: Side view of tabletop set up of methane dilution system with methane sensor housing units.

The methane sensors require significant work to set up the power and ProCoDA connection. The sensors require 5V power supply and an ethernet cable connec-

tion to plug into the ProCoDA box to record data. Figure 14 details the wiring necessary for the methane sensors. Power cord and ethernet wiring was done with a soldering iron. The resistor was also attached directly with a soldering iron, with the grounded end connected to the power supply and ethernet ground wires via a wire crimp. The prototype methane sensor used for calibration tests was constructed with a $10\text{ k}\Omega$ resistor, while the four sensors currently deployed were constructed with $10.9\text{ k}\Omega$ resistors acquired from the CEE computing office.

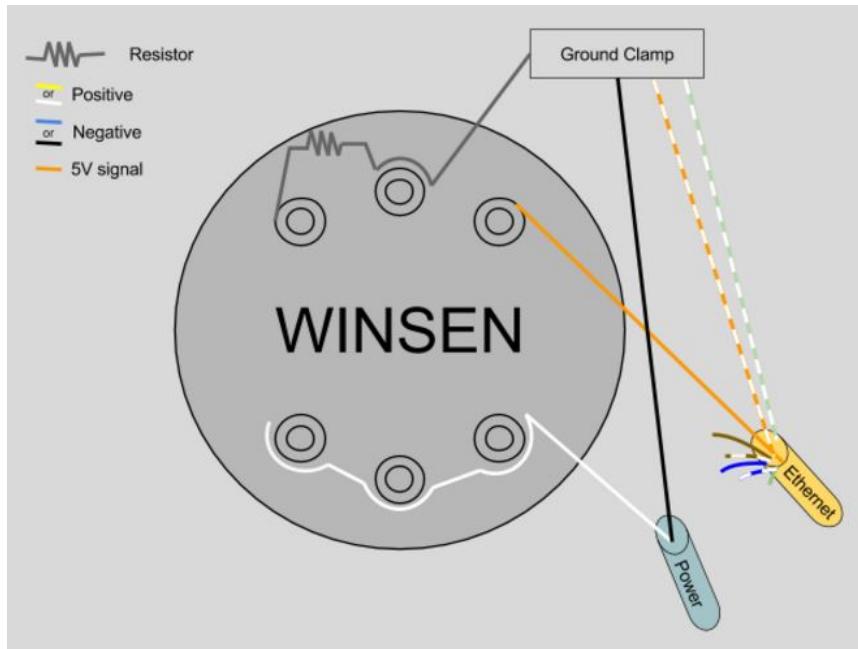


Figure 14: Diagram of methane sensor apparatus with labeling for necessary wiring connections.

2.4.2 Wastewater Preparation and Delivery

The air dilution tubing in the three stop pump, the water delivery, and the wastewater stock delivery were all tested to quantify flow rates. The air dilution was set up using purple-white 3-stop tubing. Flow rate was tested by filling a graduated cylinder with water and then inverting it. The air tubing was inserted so that air would rise through the tubing and push the water level down. It was determined that the air flow rate at 1 rpm was 0.39 mL/min . Figure 15 shows the test design.

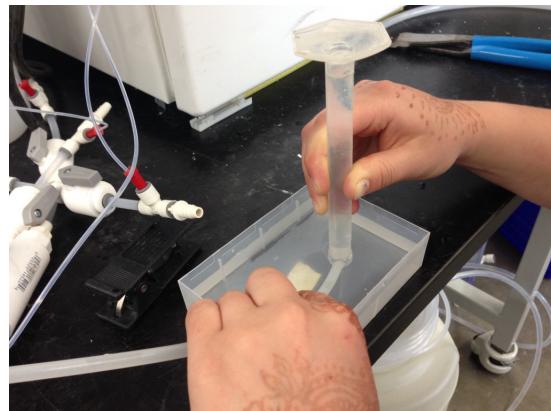


Figure 15: Flow rate test for methane dilution lines.

Testing of water and wastewater delivery was conducted to determine the ratio between delivery. The water, wastewater, and waste evacuation lines were all placed on the same peristaltic pump head so that changes in pump speed would change them all the same by ratio.

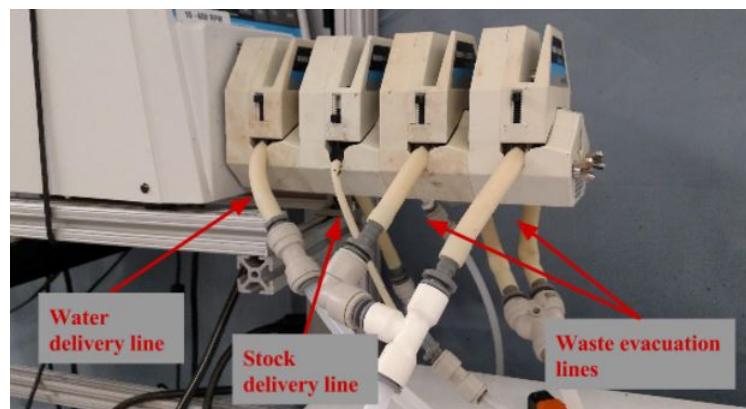


Figure 16: Peristaltic pump head supporting water, wastewater, and waste evacuation lines.

The pump set up is to test the ratio delivery between the size 18 water tubing and size 14 stock tubing, the pump was run for a certain amount of time, and water delivery was measured from both. After 3 tests, the average size 18 to 14 tubing ratio was 15.6 to 1. This stock dilution was determined to be too low, because the flow rate of 60 mL/min was faster than anytime before, and a 4 L batch of stock would last less than a day of operation. As a result, the stock should be more concentrated so that it could be pumped more slowly and last longer. Size 18 to 13 tubing ratio was tested and after 3 tests the average ratio was determined to be 70.57 to 1. Size 13 tubing required 4.52x concentration of the original stock recipe.

Table 5 includes the wastewater stock concentrate recipe that the team used during reactor operation.

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

Table 5: Concentrated Wastewater Stock Recipe

3 Inoculation and Post-Inoculation Problems

3.1 Inoculation

Before inoculation, the team realized there was no way to drain the reactor without draining the sludge. So valves were added just under the unions. (Figure 18) The sludge taken from a UASB reactor of Anheuser-busch company in late fall 2015 was used for inoculation. The sludge was loaded into the reactor through the opened union with a funnel.

Then, the reactors were filled separately from the bottom with wastewater. Originally the reactor was filled up to the union. Then, a 60 mL/min flow rate was applied. The granules clumped together and did not easily fluidize, which led to high headloss and several times of overflow. However, once the granules were fluidized, the headloss went down and became stable. At 60 mL/min , the bed expanded to around the elbow and stabilized. The granules in around 40 to 50 cm from the bottom were not obviously fluidized, while those in the upper space were just fluidized. The headloss between reactors was measured to be 3.5 cm. (Figure 17)

However, after 1 hr of operation, the granules clogged in the 1st and 4th reactors (Figure 18). The bulk granules was pushed up into tube settlers and came out of t-joint. The teams used a rubber mallet to dislodge the granule clumps. (Figure 19).



Figure 17: Headloss after inoculation



Figure 18: Clogging in the reactor



Figure 19: Clogging in the Reactor and Dislodging

The reason for clogging was unknown. But the team decided to increase the upflow velocity to prevent clogging. It was assumed that the biogas was causing granules to lock together and rise up. If the bed was all fluidized, the gas transport to the top of the reactor would be facilitated, and thus prevent clogging.

Because increasing upflow velocity means more headloss, and the water level in the 1st reactor was nearly at the top of gas collector (Figure 17), the team drilled holes about 3 cm away from the wye joint on the tube settlers to move the exit down so that more headloss could be acquired in the upper space (Figure 20). With these design modifications, the water level in the 4th reactor was forced to remain at the new exit, and the water level in the other 3 reactors rose up according to the headloss.



Figure 20: Iteration 3 Exit Design

The team increased flow rate from 60 mL/min in a 10 mL/min interval. Clogging kept occurring after a short run time. Finally, 110 mL/min flow rate was run for 8 hr without clogging. However, at this flow rate, the granules within 5 cm range at the bottom of 2nd, 3rd and 4th reactor were still not obviously fluidized. The 1st reactor was fluidized better due to pulsing from the

peristaltic pump. To be safe, the team increased the flow rate to 120 mL/min , at which all granules were fluidized. The bed was not clogged through an overnight test. In this case, the HRT was reduced to 31.7 min . So the team concluded the minimum flow rate should be 120 mL/min , equivalent to 3.58 mm/s upflow velocity. Initial fluidization tests suggested that 60 mL/min would be sufficient. However, as tests showed, the bed was not properly fluidized until 120 mL/min . The team believes that this discrepancy could be explained by the fact that different amounts of granules were used for the pre-test as the amount of granules actually used for reactor inoculation.

Because the water height could not be measured from gas collectors directly due to 1 in to $1/2 \text{ in}$ bushing, the water height was measured in tube settlers and then converted to vertical. (Figure 20) At a steady state, the water height from the 1st reactor to the 4th reactor was 13.5 cm , 9.0 cm , 3.8 cm , 0 cm . The headloss between reactors was 4.5 cm , 5.2 cm , 3.8 cm from the 1st to the 4th one. It agreed well with the sludge bed height. The higher sludge bed, the higher headloss in the previous reactor.

The figure 21 highlights headloss between reactors at different flow rates.

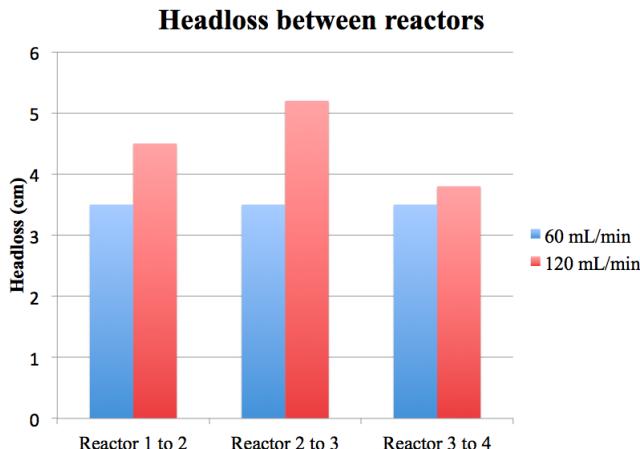


Figure 21: Headloss between reactors

3.2 Scaling Problem and Solutions

Granule clogging was the primary problem of the semester. Granule clogging led to reactor overflows, which made operation and experimentation impossible. Even at 120 mL/min , the granule bed would separate into plugs throughout the reactor, causing major headloss and overflow problems. Clogging is primarily understood as a problem of scale because the team's reactors had a small diameter of 1 in , which was chosen to allow high upflow velocity. However, small diameter reactors make plug clogging more likely to occur due to high surface tension. In larger reactors, plugs are more likely to break because weaker surface tension allows gravitational force to break up the plug itself. However, at lab scale operation, clogging has proven to be a significant challenge. It is possible that the relative amount of energy required to break up granule plugs compared to the flow rate through the reactor has increased past the point of inhibiting

reactor flow. Granule buoyancy also impacted granule rising and clogging. Bio-gas trapped within granule clumps help plugs rise and clog, presenting another operation issue. Buckingham Pi scaling calculations may be able to give some insight to whether or not the problem is strictly due to edge effects or if more complex hydrodynamics or granule biology.

3.2.1 Reactor Tilt

To address reactor clogging, the team tilted the reactors about 5 degrees to the left, as shown in Figure 22. This was done to create preferential flow along the top side of the reactor. The preferential flow decreased the prevalence of plug clogging because the granules rose on one side and settled on the other. Gas also followed this preferential flow along the top side of the reactor.



Figure 22: Tilted Reactors

However, while the tilting did help decrease plugs, plug still did form, which required frequent dislodging with the use of a mallet.

3.2.2 Barbed rod

Using the mallet to dislodge clogs had limited use but it was very time and labor intensive. To address this issue, the team worked to design another way to dislodge plugs. The barbs were made of 1/4" tubing cut into 2 cm segments. Holes were drilled on the tubing before cutting apart. (Figure 24.) Then the barbs were punctured onto PVC welding rod with around 9 cm interval. The

barbed rod in 3rd and 4th reactor were glued and those in 1st and 2nd reactor were not. One has to continuously swirling the rod when putting it in and out of a reactor because the direction of barbs were random. (Figure 23)

The barbed rods worked as both a passive and active plug agitator. Once inserted, the barbed rods extended the entire length of the reactor, with the barbs distributed along the rod in a semi-uniform manner. The barbs were supposed to work passively by attempting to break up plugs as they rose past them. However, what was more commonly observed was that the barbs kept plugs from moving up the length of the reactor by holding plugs in place. The active use of the barbed rods was the ability to pull on the top of the rod, which extended out of the top of the settling arm, to agitate the entire bed and break up plugs.

However, when the rod was externally agitated, washout was likely to occur because the barbs actually create jets throughout the reactor and therefore slow down the settling process of granules. Once granules rose up, it took a much longer time for them to settle down compared with using a single rod. Washed-out granules led to additional clogging problems in the tubing lines between reactors. So, external agitation should be done temperately and carefully.

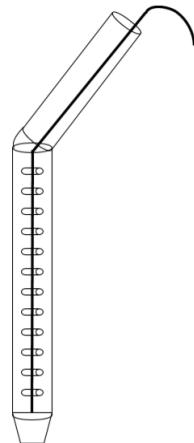


Figure 23: Barbed rod that extended along length of reactor to facilitate plug destruction.



Figure 24: Barbed rod fabrication

Plugs would rise up until it reached a barb, but the barbs were only sometimes effective at breaking apart the plugs. This led to the creation of multiple, short plugs that broke apart easily with external agitation of the rod, as shown in Figure 25.

Biogas production aided in the destruction of plugs. As shown in Figure 25, there were bubbles inside plugs. As bubbles grew bigger and made their way up through gaps between granules, plugs were broken apart. But biogas, as shown in later sections, was not created uniformly between reactors. As a result, the plugs in the 4th reactor was much more frequently agitated and destroyed, while the plugs in the 2nd reactor almost never broken up without manual agitation.

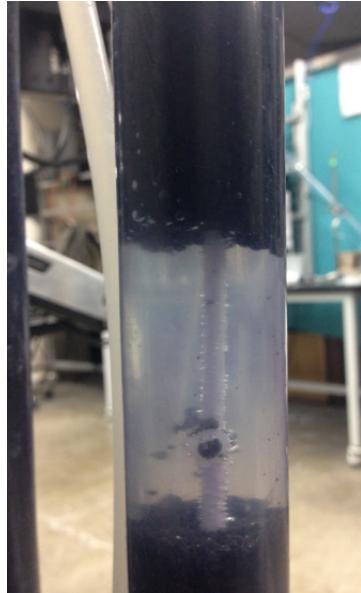


Figure 25: Barbed rod and small plug separations created by bed rising.

Once the barbed rods were inserted, the reactors were straightened from their tilted position. It was observed that tilted reactors caused "short-circuiting," which led to only partial fluidization of the bed. The top portion of the tilted reactors appeared to be moving while the settling side had either downward velocity or very little movement at all. Straightening the reactors would support more equally distributed flow and fluidization.

With the barbed rods, the reactors were able to run at 120 mL/min without major overflow problems. Due to this operational success, the team tried lowering the flow rate back down to 60 mL/min which was the original operation goal and allowed for greater HRT. The reactors ran in this lower flow rate and upright state for 2.5 days before clogging again. Plugs and headloss were attributed to be the mode of failure. The barbed rods did not successfully break up the plugs enough to eliminate failure.

In summary, the barbed rods decreased the amount of rising plugs further, but could not stop the forming from plugs. The plug can be broken by agitation from gas production, or stay under a barb with a flow bypass created beside it. But if these did not happen, the headloss would still build up because of the

plug and eventually cause overflow.

3.2.3 Recycling

After repeated clogging failure, the team tried to find a way to restart the reactor safely. So, the team added a recycle line with an additional pump pumping at 60 mL/min . It was expected to increase the flow rate without increasing organic loading, so as to prevent serious pH dropping in 1st reactor.

The recycle line was separated from the effluent right below the exit of 4th reactor. And it was designed to receive effluent with priority over the waste line. The recycled flow was combined with influent before entering 1st reactor. Thus, because the influent flow rate was 60 mL/min , the overall flow rate over the reactor became 120 mL/min , which fully fluidized the granules.

However, the reactors only ran for one hour before overflowing. The direct failure mode was sludge rising in the 1st reactor, entering the tubing between 1st and 2nd reactor and causing huge headloss. It revealed a huge problem in recycle line design. Once clogging or headloss accumulation reduced flow, effluent from the 4th reactor was reduced and the recycle line started sucking air and aerating the 1st reactor. Then, the sludge rose up from 1st reactor and clogged the tubing between the 1st and 2nd reactors, which in turn, caused more overflow and aeration.

Apparently, the recycle line was carelessly designed. But it rose the question that whether a recycle line can be safely added into the system. Because the reactors was an open system, if a recycle line is to be added in the future, there much be some mechanics to prevent potential aeration in the 1st reactor.

3.3 Acidification

Acidification had been a major problem to determine if the reactors could be run sustainably. In kinetics, this problem is due to the two-stage reaction of anaerobic digestion. In the first step, the fermentative acidogenic bacteria convert biodegradable organic matters (e.g. carbohydrates, proteins, lipids) into acetate, hydrogen and carbon dioxide. In this process, pH might drop if alkalinity is not sufficient. In the second step, the methanogens convert the products from 1st step into methane. The methanogens are very sensitive to pH. The optimum pH for methanogens to function is 6.6 - 7.6 and outside of this range, methane production is inhibited. At pH=6, methanogens would die. If the pH drops too much in a single reactor, the methanogens would be inhibited or even die out. There are usually two remedy for this problem, that is, reducing organic loading, so as to reduce the acid production rate, or adding alkalinity in wastewater to consume the protons.

During the operation of the reactors, the team tested the pH in the reactors several times, especially the first one. On April 7th night, the team first tested the pH in 4 reactors with pH strips, when the reactor had been running at 100 mL/min , tilted, for 2 days. (Figure 26) The samples was taken from the valve above reactor union very slowly to minimize the impact on the flow. The pH was between 4-5 for the 1st reactor, and 6-7 for the 2nd one, and 7 for the 3rd one and 4th one. And the least methane was collected from 1st reactor as well.



Figure 26: Results from pH test April 7th (strips from reactor 1 through 4 from bottom to top respectively)

To solve this problem, the team reduced the stock concentration by diluting it from 2 L to 3 L on April 28th. After about 15 hours, when the team tested the pH in the 1st reactor again. It rose up to 7, measured by both pH probe and pH strip. It is assumed that the reduced organic loading led to the pH recovery. To confirm this problem, the team switched back to normal stock concentration again to see if pH would drop again. On May 1st, the pH in 1st reactor was 6-7 when it was just restarted. On May 7th, the pH in 1st reactor was 7 after 2 days running with barbed rod and on 60 mL/min. However, after the first test, the reactor was faced with many other problems, including multiple times of granule clogging, stock feeding line clogging and stock characteristic change due to unavailable autoclave. As a result, the flow rate was changed frequently, and barbed rod was put into the reactors, all affecting the organic loading of the reactors. So, it was hard to tell the actual cause of pH change.

However, later, the team found all results from pH tests skeptical, because water coming out from the valve could be acidic at first and then approximately neutral. So the sampling method might be wrong. And the pH strips were not accurate measurements either. But because the appearance of granules in the 1st reactor was always not as healthy as others, especially after long time of continuous running, and the fact that almost no methane was ever produced in the 1st reactor, the concern of acidification was not completely unfounded. Closer pH monitoring with pH probe inside the reactor should be developed in the future.

4 Experimental Data

COD measurements and methane production were the two experimental data that the team was able to collect during the reactor's short operational period. Small amounts of data were collected because the teams efforts were primarily

focused on declogging, but the samples gained offer some insight into the functioning capacities of the reactors. COD removal was documented, and methane production was well observed and recorded.

4.1 COD Tests

A COD test was done to understand COD removal when the reactors were running at 60 mL/min with full strength wastewater stock. On May, 7th, samples were taken at influent, between each reactor, and at effluent for a total of five samples. The samples were taken from the top of the settling arm for each reactor, but accidentally included some rising solids. To remove the solids, the samples were centrifuged for 8 minutes at 2500 rpm. Two milliliters of each sample, without solids, were added to prepackaged vials from potassium dichromate CHEMetrics packages. Two COD vials were used for each sample to get an average COD from each effluent. The vials were incubated for two hours at 150 degrees Centigrade, and then tested colorimetrically by a Hewlett Packard Diode Array Spectrophotometer.

The results from the COD test are shown in Table 6.

Sample	Min Concentration (mg/L.)	Average Concentration (mg/L.)	Max Concentration (mg/L.)	Percent Removal (compared to influent)	Percent Removal (compared to previous reactor)
Influent	341	361	381	0	0
Reactor 1	342	348.5	355	3.46	3.46
Reactor 2	283	293.5	304	18.7	15.8
Reactor 3	277	305.5	334	15.4	-4.09
Reactor 4	223	223.5	224	38.1	26.8

Table 6: Minimum, average, and maximum COD Concentrations with percent removals.

To get percent COD removal across the entire reactor series, final average concentration was divided by initial average concentration and then subtracted from 100, and multiplied by 100:

$$(100 - \frac{223.5}{361}) * 100 = 38\%$$

A clear trend of decreasing COD levels across reactors is highlighted in Figure 27. The percent removal from reactor 3 as compared to reactor 2 is a negative number, which is attributed a number of different errors because COD cannot increase. One error is the fact that substrate within the reactors is not uniformly mixed, so there are parts of low and high concentrations. The reactors functioned closer to a plug flow reactor (PFR) than a completely mixed flow reactor (CMFR). Another error explanation is that the samples create trend lines that have error bands that associated with them; the trend is downward, but possible values exist within an error band that might be higher or lower than the value documented by the COD test. The samples used were only from one day and taking more samples would help correct for these errors.

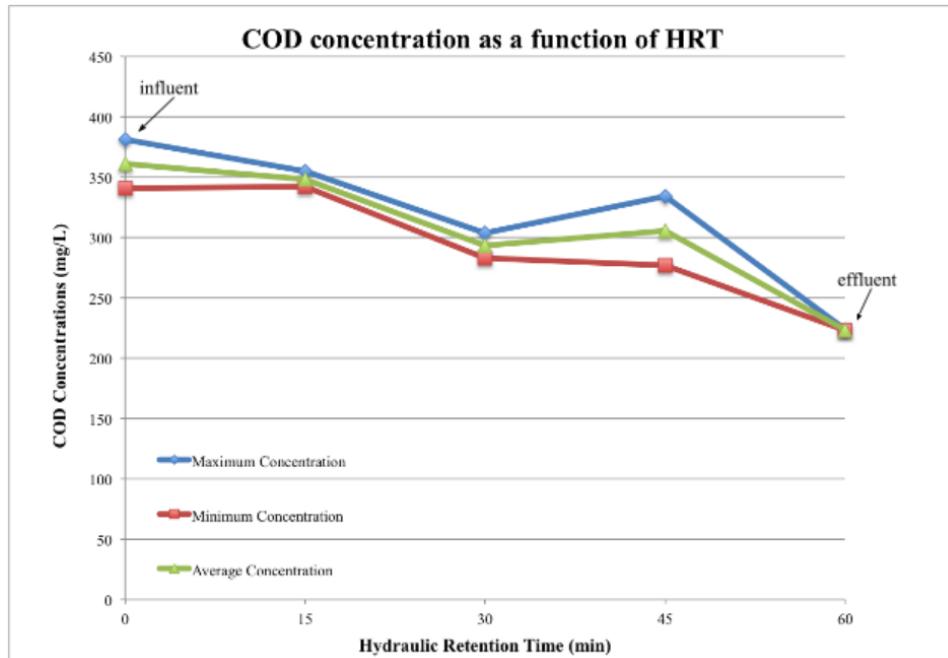


Figure 27: Minimum, maximum, and average COD Concentrations vs HRT.

4.2 Methane Production

On April 27th the team constructed the first methane sensor and started collecting data from the 2nd reactor. On May 2nd, all 4 methane sensors were established and started collecting data. The data was collected until May 9th, in the unit of volts. With the correct calibration curve, the methane sensor results will be displayed in units of volumetric methane flow rate. However, in the fabrication of methane sensors, the team used a different resistance from the one based on which the former calibration curve was produced. A new calibration curve was needed in the future to convert the data in volts into methane flow rate.

Due to occasional ProCoDA data collection failure and the difficulty of keeping methane concentration within detection range. The team frequently adjusted the air flow rate in order to keep methane data in range until May 7th, when the pumping speed was finally increased to 100 rpm. The corresponding flow rate was 38.2, 37.2, 42.6 and 34.7 mL/min for reactors 1 to 4 respectively. And the reactor was clogged in May 8th, and stopped running on May 9th. Thus, the only piece of continuous and mostly in range data was collected from May 7th 14:00 pm to 8th 07:43am (partially shown in Figure 28).

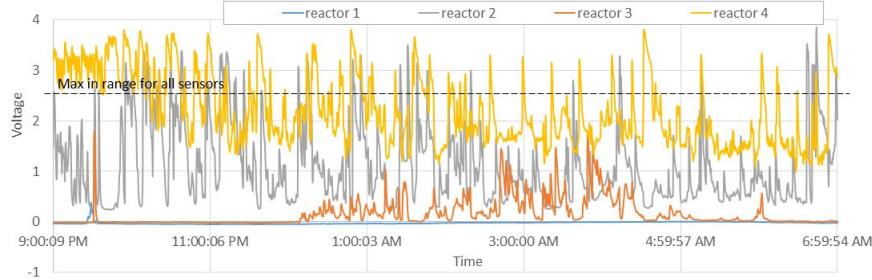


Figure 28: Methane sensor data collected from May 7th 9:00 pm to 8th 07:00am with 5 seconds interval

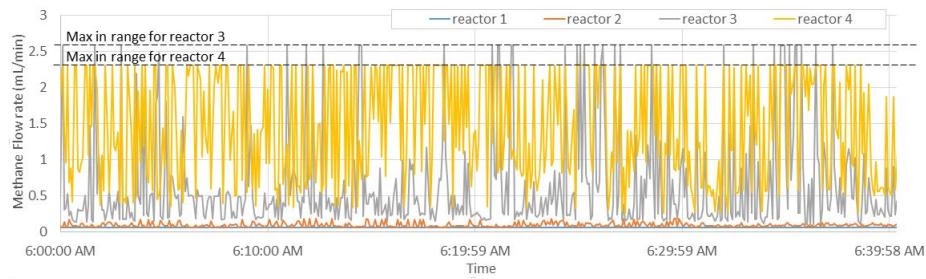


Figure 29: Processed Methane sensor data collected from May 8th 06:00am to 06:40 am with 5 seconds interval

The data in Figure 28 was collected from May 7th 9:00 pm to 8th 07:00am with 5 second interval. According to the original calibration curve in figure 11, the linear range for voltage data collected directly from methane sensor should be below 2.6 volts. Thus, half the data for 4th reactor was still out of range. To estimate the approximate volumetric methane flow rate, set all data above range to be 2.6 volts, and apply the original calibration curve to get methane flow rate.

Figure 28 shows the 4th reactor has the most methane production rate, and the 3rd one and 2nd one. The 1st reactor was barely producing any methane.

Daily COD removal:

$$361 \frac{\text{mg}}{\text{L}} \cdot 0.381 \cdot \left(60 \frac{\text{mL}}{\text{min}} \right) = 11.884 \frac{\text{gm}}{\text{day}}$$

Estimated daily COD removed (Typically 350 mL (STP) CH₄ is produced per 1 g COD destroyed for domestic wastewater):

$$\frac{350 \text{mL}}{\text{gm}} \cdot \left(11.884 \frac{\text{gm}}{\text{day}} \right) = 4.159 \frac{\text{L}}{\text{day}}$$

Assume effluent is saturated with methane (solubility 0.023 g CH₄ /kg water at 20 degree celcius), daily methane discharged in liquid phase:

$$\frac{0.023 \text{gm}}{\frac{\text{L}}{16 \frac{\text{gm}}{\text{mol}}}} \cdot 22.4 \frac{\text{L}}{\text{mol}} \cdot 60 \frac{\text{mL}}{\text{min}} = 2.782 \frac{\text{L}}{\text{day}}$$

Theoretical methane daily production in gas phase:

$$4.159 \frac{\text{L}}{\text{day}} - 2.782 \frac{\text{L}}{\text{day}} = 1.377 \frac{\text{L}}{\text{day}}$$

Measured methane daily production in gas phase (based on original calibration curve set the maximum value to peaks that are out of range):

Methane in gas phase (mL/day)	Reactor 1	Reactor 2	Reactor 3	Reactor 4	Total
99	165	865	2298	3426	

4.3 Granule Characterization

Following a final reactor overflow, the sludge was allowed to settled for a day. Sludge samples were then taken and characterized for size using the same dish sample size and grid paper as the granule pre-test. The results are displayed in Figure 30.

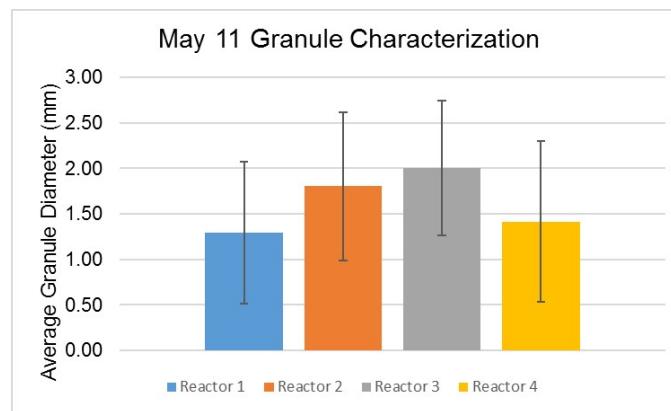


Figure 30: Granule Diameter Sample Mean and Standard Deviations from settled reactors, taken on May 11

Although the standard deviations of the sample size taken are high, it appears that in reactors 1 and 4 there was a significant decrease in granule size from inoculation. The mean granule sizes observed in the first and last reactors were 1.29 and 1.42 mm respectively, both almost a full standard deviation lower than the initial granule diameter of 2 mm. This indicates that the larger granules are likely being broken up or washed out of these two reactors. While this is not unexpected for the first reactor, which was having acidification problems, it is unexpected for reactor 4 which appeared to be producing significant amounts of methane. It is possible that the broken up granules washed out of the previous reactors have accumulated in reactor 4, but this would imply that the wash out velocity of reactor 4 is lower than the preceding reactors. This could be due to accumulated headloss, but further investigation should be done before making this conclusion. The mean granule sizes in reactors 2 and 3 were 1.80 and 2.00 mm respectively, close enough to the initial mean granule size that significant change cannot be said to occur.

Conclusions

Despite both hydraulic clogging and biological failure in the first reactor from acidification, the bench-scale EGSB was observed to have both significant methane production and COD removal. This indicates that if the flow obstructions and acid production can be optimized with reactor modifications and operational adjustments to organic and hydraulic loading rates, the data collected could lead to a larger scale pilot system. The main purpose of such a pilot would likely be to optimize methane production rather than COD removal, as the small villages that AguaClara works with generally do not have BOD issues. So long as the high rate system is capable of producing useful quantities of methane at a cheaper capital cost than a more conventional system, a partial COD removal rate is potentially acceptable.

Future Work

Methane Production

The team wired and attached four methane sensors to the top of each reactor to gather the produced methane. Data was collected in voltage form through ProCoDA, and can be processed to volumetric methane flow rate given atmospheric pressure and total volumetric air flow rate across the sensor. However, the sensors have a very narrow range of methane concentrations and the recorded voltage often rose out of the measurable range during reactor operation.

Methane Sensing

The methane sensors need to be recalibrated to account for the changed resistor size. In addition the dissolved methane in the reactors needs to be accounted for in the calculation of total produced methane. This is because of the low strength of municipal wastewater relative to typical industrial wastes fed to anaerobic reactors. The calculation will be based on Henry's Law, and require estimates of the partial pressure of methane present in the reactor headspaces, surface

areas of each air-water interface, and an estimate of total methane production based on COD destruction. Additional work should also be done to work on a better system of air dilution. When biogas rises in large bubbles, the readings are outside of the methane sensor ranges and does not report good data. A possible way to address this is to figure out a way to sense a bubble as it comes and then change the dilution pump accordingly. External agitation would make bubble release more constant, as highlighted in later sections. With more constant bubble release, a longer sampling frequency could be used because there should not be large episodes of methane release.

In future semester, dilution lines should be equipped with their own pump heads so they can be manipulated individually. Ideally the dilution flow rate in each line or set of lines would be dynamically adjusted to keep the methane concentration within measurable range. Such a system would require at least one ProCoDA controlled pump that is programmed to respond to changes in sensor voltage. Data showed that the 1st reactor produced almost no methane while the 3rd and 4th produced a lot. Separating at least the first reactor's dilution line from the following three would improve the accuracy of methane measurements recorded by the sensors.

Stock Delivery

Size 13 stock delivery lines were adopted to work with 4.5x concentrated stock. Clogging issues in the stock lines required frequent monitoring to ensure effective delivery of stock.

Barbed Rod Re-Design and Mechanization

The current barb design using welding rod and $1/4\text{ in}$ tubing presents issues with plug creation. As discussed above, the barbs prevent the plugs from floating very high, but does not prevent gaps from forming. It is possible that the barbs also prevent the granules from falling down easily, because they get caught on the top of the barb. A re-designed barbed rod system could prevent plugs from rising by disturbing the plug shape, but also support the settling by having a more pointed top. A proposed redesign is shown in Figure 31. Future tests with barb design could help determine whether or not the barbed rod can be effective at preventing plug caused clogging.

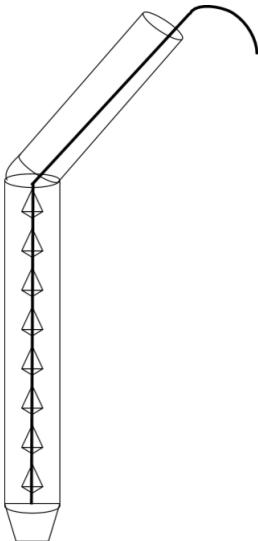


Figure 31: A proposed barbed rod re-design, with barbs shaped as diamonds rather than cylinders.

Another option to optimize barbed rod efficacy would be to mechanize a way to move the rod at a constant rate to physically break up plugs. Plugs break up well when the rod is pulled and pushed continuously; however, if the rod is not moved continuously, then plugs do not disappear and simply move up the reactor to the next barb. Additionally, the agitation cannot be too forceful, because then significant granule washout occurs. Agitation in the form of a pulse might be the most effective way to break up granule plugs. During operation, the first reactor was observed to be pulsing due to the variable pumping from the peristaltic pump. The first reactor also had the least clogs, so if other reactors could be pulsed similarly with the barbed rod then clogging problems might be solved. A solenoid valve controlled with ProCoDA and connected to the top of the barbed rod could pulse the entire thing. Future tests need to be done to determine the actual force required to break up the plugs.

Another benefit of reactor agitation is the ability to break up large biogas bubbles into smaller ones. When biogas is trapped or inhibited to rise by plugs, it makes it difficult for biogas to rise as it is created. If the reactors were constantly agitated, it would make it easier for biogas to escape as it is being created, which would limit the trapping of bubbles and eventual rising of large ones. Large bubbles saturate the methane sensors, and cannot be diluted by the dilution pump effectively. Smaller bubbles from agitation are easier for the methane sensor to read and process.

COD Removal Efficiency

A second COD test was conducted on just influent and effluent. The results from the test are shown in Table 7. The COD removal from influent to effluent was 84%, which is significantly different from the previous COD test that showed 38%. It is clear that more frequent COD tests need to be done to account for

reactor nonuniformity, testing error, and operational changes.

Sample	Min Concentration (mg/L)	Average Concentration (mg/L)	Max Concentration (mg/L)	Average Percent Removal
Influent	177	180	183	84
Effluent	14.8	28	41.2	

Table 7: Minimum, average, and maximum COD Concentrations

As experiments determine optimal reactor flow rates, frequent COD tests should be done to better understand the impact of HRT on COD degradation. If the original 38% removal after 1 *hr* HRT data is extrapolated from, it can be suggested that increasing HRT to 2 *hrs* could decrease COD concentrations to as low as 80% of initial concentrations. However, the second COD test with 84% removal after 1 *hr* might suggest better COD removal than originally accounted for.

pH Monitoring

Due to the fluctuating headlosses of the reactors, a continuous probe measurement of pH has not yet been attempted. As a result, it is hard to pinpoint the cause of the first reactor's acidification. The team has discussed a possible design for a sampling pipe attachment to the effluent of the first reactor, into which the lab pH probe could be connected. This would protect the probe's wiring from changing water level, as well as allow for the easy removal of the probe using an upstream valve.

Reactor Modifications

Two design modifications could significantly improve reactor operation: a new 60 degree settling arm and headspace maintenance ports. The current reactor design has a 45 degree settling arm, which was chosen because wye joints only come at 45 degree angles and with the previous pressure-sensor gas collection method, a level headspace was required. However, with new AguaClara ability to mold PVC pipe and new methane sensors that do not require level headspace, and 60 degree settling arm could be obtained. This would increase granule settling capacity, which would decrease the amount of washout. Additionally, plugs formed in the settling arm would be decreased because they would be forced to fall back down into the main portion of the reactor.

There were also issues with granules rising and getting stuck in the methane collection chambers. It is unclear if granules in the headspace might cause future problems by limiting the ability of methane to flow to the methane sensor. However, adding a way to clean and maintain that portion of the reactor would be beneficial. A union to connect the settling arm to the gas collection chamber would allow for easier maintenance and cleaning, without having to break down the entire reactor.

References

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Semester Schedule

Task Map

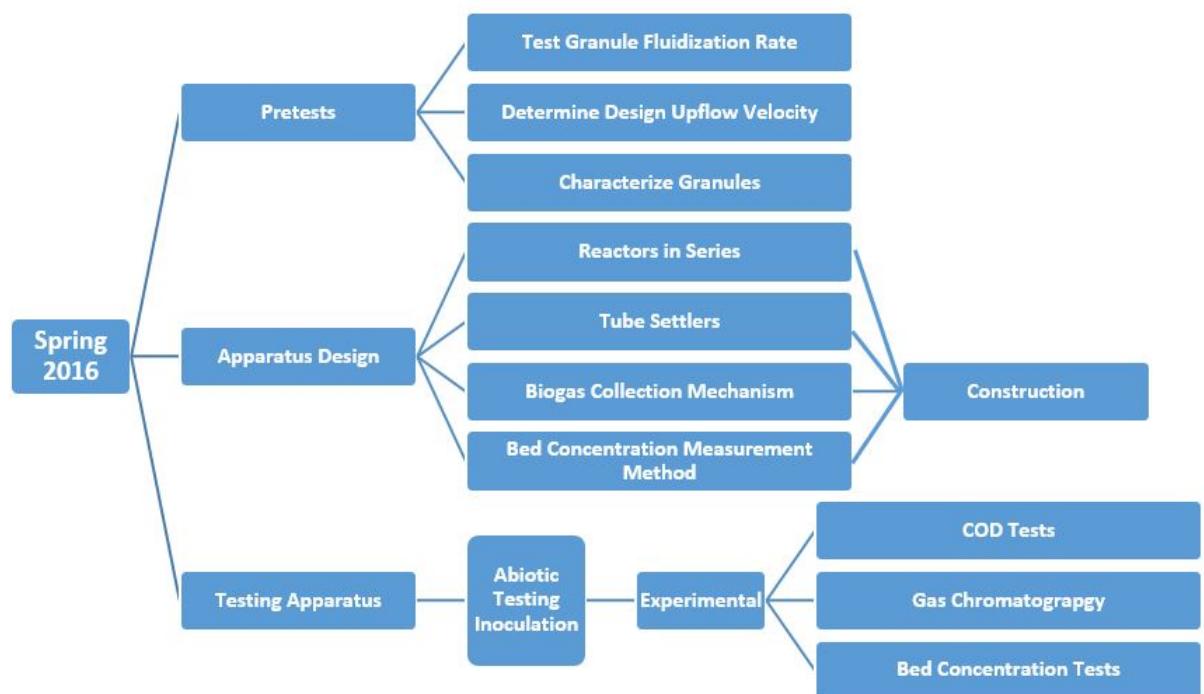


Figure 32: HRUASB Spring 2016 Task Map

Task List

Pretests

Fluidization tests and granule characterization/Feb. 19 - Qiu. Complete

1. Test granule fluidization at different upflow velocities in 1-inch clear pipe using cone inlet jet system. 2/19 - Complete
 - (a) Measure fluidization by percent bed height expansion.
2. Determine the optimum upflow velocity for fluidization and plate settler design. 2/19 - Complete
3. Characterize granules to determine typical size range. 2/19 - Complete

Design

Design of new EGSB reactors/Mar. 4 - Zoe. Design new reactors with higher flow rates, lower HRT, and higher SRT.

1. Determine reactor specifications to meet necessary retention times with optimum upflow velocity. 2/29 - Complete
2. Create process map to include: (2/29) - Complete
 - (a) Flow of influent and effluent
 - (b) Sampling ports
 - (c) Process Controller connections
3. Design items to include: (3/2) - Complete
 - (a) Arm settlers
 - (b) Reactors in series
 - i. 4 reactors in series, each with inlet and effluent regions that can be sampled
 - (c) Biogas collection mechanism
 - i. Air-tightness and pipe connectivity
 - ii. Process Controller off-gas code
 - iii. Calibrated pressure sensors
 - iv. Valves to allow off-gas events
 - v. Septa for gas chromatography tests
 - (d) Method to measure bed concentration at different depths
4. Present design options to Monroe (meeting week of 2/29), and choose design specifications (3/4) - Complete
5. Order parts - update needs on spreadsheet throughout work

Construction

Construct the UASB reactor/Mar. 16 - Stephen. Complete

1. Use fabrication techniques developed by previous wastewater and rapid sedimentation teams to construct a tabletop experiment. 3/2
 - (a) Figure out tabletop reactor stands.
2. Organize reactors, Process Controller, and refrigerator to make sure all components fit in tabletop experimental system. 3/7

Abiotic Testing

Abiotic reactor testing - Qiu.

1. Test equipment to ensure experimental set up is consistent with process map plans. - Workin on
 - (a) Reactor Iteration 1: construction according to first design
 - (b) Reactor Iteration 2: design modifications to account for reactor filling and reactor operation flaws with overflow and headloss issues.
 - (c) Reactor Iteration 3: address design flaws from Iteration 2 that caused reactor overflow and creation of sludge plugs.
2. Test methane sensors

Inoculation

Inoculate - Zoe. Complete

1. Autoclave container and make wastewater stock. Complete
2. Fill all 4 reactors components with same amount of granules (about 1/3 of reactor height), and begin feeding. Run pumps at predetermined optimal flow rates. Continue to check that reactors are being fed successfully.

Experimental

Tests will be run from inoculation until reactor shut-down at end of semester.
Complete

1. COD tests.
2. Methane Sensor tests.

Analysis and Report Writing

1. Analyze experimental tests to determine success of reactor design and experimental operation.
 - (a) Compare HRUASB results to previous UASB team results
2. Report writing and presentation.

Report Proofreader: Stephen Galdi

Operating Notes

Date	Note	Failure		Operation parameters			pH test	COD test
		stock	reactor	Water flow rate (mL/min)	Air pump speed	Others		
20-Apr	innoculate		clogged and dislodged	60		stock feed tubing 14		
21-Apr	reactor modification			120		stock feed tubing 13		
22-Apr		clogging		120		stock filtering		
23-Apr	no problem			120				
24-Apr	granules in reactor 1 greying, Observed bubble production 4>3>2>1		clogging and turned off	100		reactor tilts 4 degrees		
25-Apr			reactor restarted, clogged again at night	100		Inserted agitation rods, tilted reactors to 7 degrees		
26-Apr	no problem			100			R1 - 4.5, R2 - 6.5, R3 - 7.0, R4 - 7.0	
27-Apr	no problem			100	2	Added first methane sensor to reactor 2		
28-Apr	concern about acidity			100	2	diluted stock from 2L to 3L		
29-Apr			clogged and dislodged	100	2	stock back to original concentration.	R1 - 7.0	
30-Apr	lost some sludge		clogged and turned off	100	2			
1-May			reactor restarted, keep clogging until increase flow rate to 110	110	2		R1 - 6.5	
2-May	no problem			100	2	All 4 methane sensors set up and zeroed		
3-May		clogged		100	2	methane sensor 3 re-zeroed	R1 - 6.5	
4-May			clogged and restarted	110	2	methane sensor 4 re-zeroed, new batch of stock no autoclave		
5-May	many bubbles in reactor 4, no methane in reactors 1 to 3, small plugs formed			60	4	Inserted barbed rod and straightened reactors, ProCoDA Daq failure		COD sample taken before changes
6-May			clogged and dislodged	60	10			
7-May			Overflow in reactor 1, turned off and back on	60	100		R1 - 7	COD test
8-May			Overflowed and stopped	60	100			
9-May			reactor restarted and quickly clogged	60	100	failed recycle		