Upflow Anaerobic Sludge Blanket, Fall 2016

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Abstract

To lower the amount of wastewater discharged into natural bodies of water, low-cost, energy-efficient treatment technologies are needed in developing countries. The AguaClara team has developed lab-scale UASB reactors to evaluate the degree of treatment and energy recovery from "black water" (concentrated wastewater from toilets). Specifically, the objectives this semester were to improve the current laboratory set-up and equipment, adjust the wastewater stock recipe to prevent clogging in influent lines, and determine the residence time and maximum organic loading rate for the reactors. Ultimately the set-up changes were effective and a sufficient wastewater stock recipe was developed. Tracer tests found the residence time of each reactor to be approximately three hours, but the maximum organic loading rate has yet to be characterized.

1 Introduction

The Upflow Anaerobic Sludge Blanket (UASB) team is researching energy-efficient wastewater treatment technologies with applications in small villages in Honduras. "Black water," which is concentrated wastewater from toilets, is rich in organic matter and provides an ample opportunity for energy recovery. Because up to 80 percent of wastewater is not treated globally and is often discharged into natural bodies of water, low-cost, energy-efficient technologies are needed (UN-, 2014).

As a result, the UASB team has fabricated four lab-scale UASB reactors to test the capacity of treating synthetic black water anaerobically. Ultimately, the goal is to push the limits of conventional anaerobic wastewater treatment and develop new ways to treat high-strength wastewater. Thus, the current capacities of well-established technologies such as UASB must be characterized. Chemical oxygen demand (COD) removal and biogas production were monitored as a measure of reactor performance. Although the ultimate goal was to observe how reactor performance changed with an increasing organic loading rate (OLR), the maximum organic loading rate was not determined this semester due to time constraints. Smaller tasks have been accomplished in order to produce successful results and characterization. Because the synthetic wastewater recipe used formed particulate matter that settled in influent lines, methods of preventing this clogging were investigated. For example, the laboratory set-up was changed to minimize the length of influent delivery lines and to increase the velocity within the influent tubing. Furthermore, the wastewater stock recipe was modified to reduce the amount of particulate matter in order to prevent biomass accumulation in influent tubes. The reactors were also repaired to establish gas-tightness. Modifying the synthetic wastewater recipe and measuring biogas production required collaboration with the Anaerobic Fluidized Bed (AFB) team and Sensor Development team, respectively. In addition to characterizing the OLR, the hydraulic residence time (HRT) of the reactors was determined using tracer studies. The characterization of lab-scale UASB reactors in treating black water is crucial to understanding how sustainable wastewater treatment technologies may operate as full-scale pilot plants in Honduras.

2 Literature Review

Aiyuk et al. (2004a) suggested that there is a large need to develop wastewater treatment systems in developing countries, and that such a task is a nontrivial undertaking. There are many constraints for widespread applications in the developing world, such as simple designs, use of non-sophisticated equipment, high treatment efficiency, and low operating and capital costs (Aiyuk et al., 2004b). UASB technology has been widely used in the wastewater industries for a number of reasons including simplicity, scalability, and the ability to process a variety of wastewater strengths (Aiyuk et al., 2004b). Thus, UASBs provide an opportunity in developing countries as a potential wastewater treatment system.

Chong et al. (2012a) provided an overview of the current state of UASB technology. The typical UASB consists of a cylindrical column and a gas-liquid-solid (GLS) separator with a geometry traditionally similar to a funnel Figure 1. Wastewater is fed into the bottom of the reactor and flows upward, and the particles separate due to differences in density. The dense sludge forms a bed at the bottom of the reactor, and the smaller particles form a blanket above the sludge bed. The reactors are inoculated with bacterial biomass that eventually undergoes a process called granulation that occurs in three main steps: absorption, adhesion, and multiplication. After inoculation and granule formation are complete, the influent COD is converted into biogas, a gaseous mixture consisting mainly of methane (CH_4) and carbon dioxide (CO_2). This biogas can then be collected and used for energy.

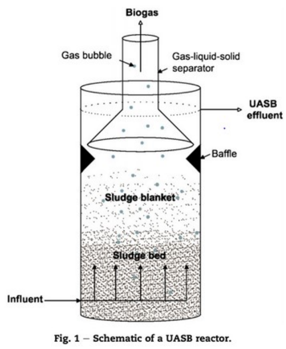

Figure 1: Schematic of UASB Reactor Chong et. al 2012

The complex microbial community within the granules converts influent wastewater into biogas via anaerobic digestion (AD) as shown in Figure 2. The AD process includes hydrolysis of non-soluble biopolymers into soluble organics, acidogenesis or the conversion of soluble organics into fatty acids and CO_2, acetogenesis in which the fatty acids are converted into acetate (or acetic acid) and hydrogen gas (H_2), and finally, methanogenesis in which the acetate, CO_2, and H_2, are converted into CH_4 gas. Acidogenic bacteria are usually responsible for the hydrolysis and acidogenesis steps, acetogenic bacteria for generation of acetic acid, and methanogenic bacteria for the final conversion into methane De Mes et al. (2003).

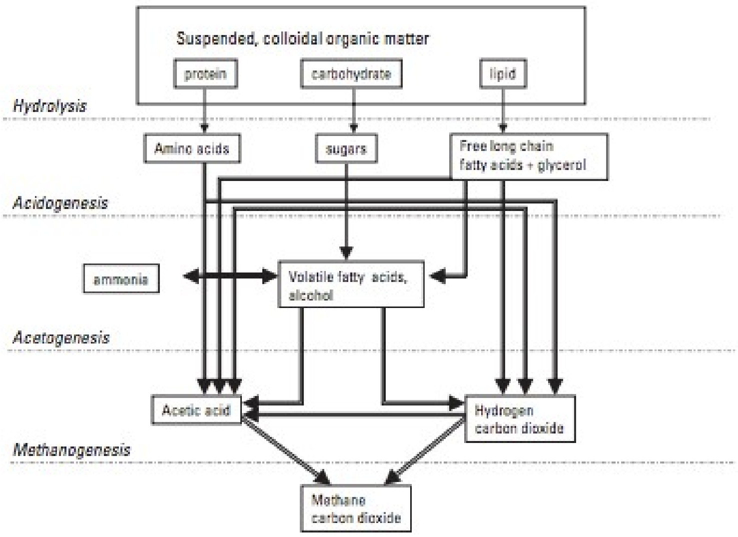

Figure 2: Wastewater to Biogas Chemical Conversion Process Flow Diagram Mes et. al 2003

Both AD and UASB are well-established technologies that have been researched extensively, including reactor start-up and granulation characterization, couplings with post-treatment units, and general improvements in operating efficiency. However, the technology faces limitations when applied in developing countries, most notable of which involves the ability of the reactor to maintain a steady performance in a fluctuating climate Chong et al. (2012b).

De Graaff et al. (2010) examined the feasibility of applying UASB technology for the treatment of concentrated black (toilet) water. UASB was effective in removing COD of the black water with high methane concentrations, but the effluent needed further treatment to remove additional COD and to recover nutrients such as nitrogen and phosphorus. Furthermore, while UASB technology may be appropriate for black water treatment, further research is needed to explore the applications in developing countries De Graaff et al. (2010), such as the fluctuating climate and costs to implement such technologies.

A comprehensive synthetic wastewater recipe (SYNTHES) was developed by Aiyuk et al. (2006). It was found that granulation decreased when using SYNTHES, but high COD removal and methane production was achieved. Various other synthetic wastewater recipes have developed for a wide variety of experiments (Cheong and Hansen, 2006) (Cadi et al., 1994). In general, there are no strict guidelines in developing synthetic wastewater. Basic components such carbohydrates, lipids, nitrogen, calcium, and phosphorus tend to be included, yet the exact constituents to incorporate these elements are widely varied (Alzate-Gaviria et al., 2007).

3 Previous Work

Dating back to the summer of 2013, wastewater research is a relatively new development for AguaClara. In the four semesters of wastewater research, the teams have made many developments, but have also run into many challenges. Starting in the summer of 2013, wastewater research began with the fabrication of two UASB reactors. Gas production and COD removal of the two reactors was tested. Support media was utilized in one of the reactors to promote biomass growth.

During the Fall semester of 2013, six more reactors were built. Half of the reactors were UASB reactors, and the other half were AFB reactors. Lengthy start-up periods and gas leakage prevented sufficient data collection on gas production and COD removal. However, this group made large strides with granule characterization. Models for biomass fluidization and settling were developed, and confocal microscopy was used to further characterize the granules.

The Spring 2014 team started to use gas chromotography (GC) as another method to measure methane from the reactors. Unfortunately, this group also had trouble with gas leaks from the reactors. Excess Teflon tape and para-film to make the reactors airtight was only successfully for one of two reactors, and thus data collection was difficult and inaccurate. This team also ran into issues with nonuniform COD delivery (Minot and Greenberg, 2016).

The Fall 2015 team with Evan Greenberg and Zoe Maisel picked up where the previous research left off. As a team of new members, much of the semester was dedicated to studying literature and gaining an understanding of the technology. Reactors were tested for air tightness, and the most airtight reactors were used for research. The lengthy start-up time and leaky reactors hindered the team research and not very much useful data was obtained (Maisel et al., 2015).

The Spring 2016 team made the decision to dedicate the semester to fabricating new reactors to combat the persistent issues associated with biogas leakage. These reactors has subtle design changes to make the maintenance and cleaning of the reactors simpler. The new design is shown in Figure 3. Four new reactors were fabricated, but no data in gas production or COD removal was collected (Minot and Greenberg, 2016).

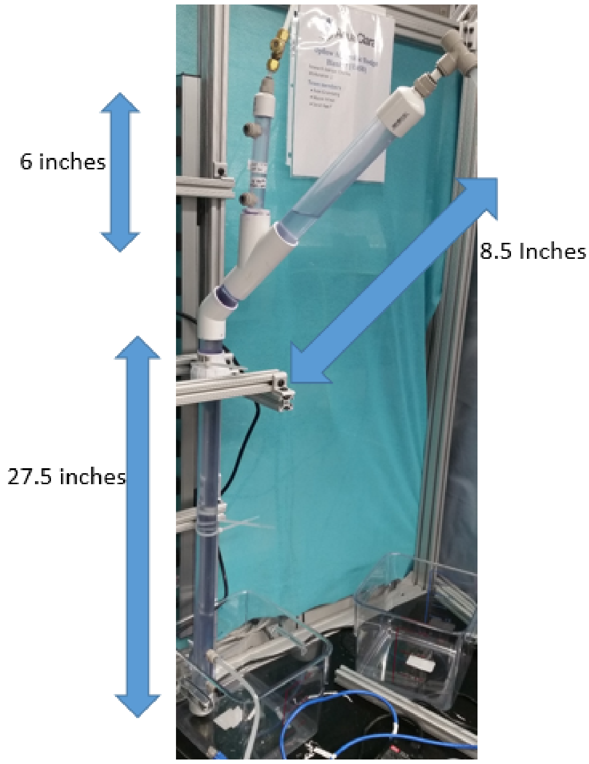

Figure 3: Prototype UASB design

Over the summer of 2016, the newly constructed reactors were utilized by Andrew Kim. Under the Engineering Learning Initiatives grant, the UASB reactors fabricated in Spring 2016 were operated. Gas-tightness in each reactor was successfully established by switching from push-to-connect connections to ferrule compression brass fittings. Systems for optimal wastewater stock delivery and methane concentration measurement were developed and led to the current set-up employed by the Fall 2016 team, which will be discussed below. High strength wastewater treatment started to be characterized, but the full limits of the reactors were not explored.

4 Preliminary Operational Changes

Before HRT and OLR tests could begin, a few preliminary changes were made. The first was to change the laboratory set-up to make operation easier. The second was to repair the headspace of Reactor 2 (R2), which was noticeably damaged. Lastly, the synthetic wastewater stock recipe was modified to reduce particulate matter formation.

4.1 Laboratory Set-up Changes

A big challenge this semester was to change the set-up to reduce the amount of biomass accumulation in influent lines. Thus, steps were taken to reduce the length of tubing that carried synthetic wastewater. A new hole was drilled in the refrigeration unit using a small diameter saw to reduce the distance the stock needs to travel. The new hole in the refrigeration unit allowed for shorter influent tubings to connect to peristaltic pumps more quickly. Pumps for the wastewater stock and water line were also moved closer to the reactors to accommodate the new set-up. The addition of a new hole in the fridge has not affected its ability to stay cool, and no additional plugging is needed. One disadvantage of this new set-up is that the tubing used to carry concentrated stock solution is now slightly longer, but the lines that carry diluted stock are much shorter. It was determined that having much shorter diluted stock lines are worth the additional length in the concentrated wastewater stock tubing, as the diluted stock lines were the primary source of biomass accumulation. Figures 4 and 5 shows a picture of the new set up. ZM: [this picture should be edited to make sure not to include the red line under microbore]

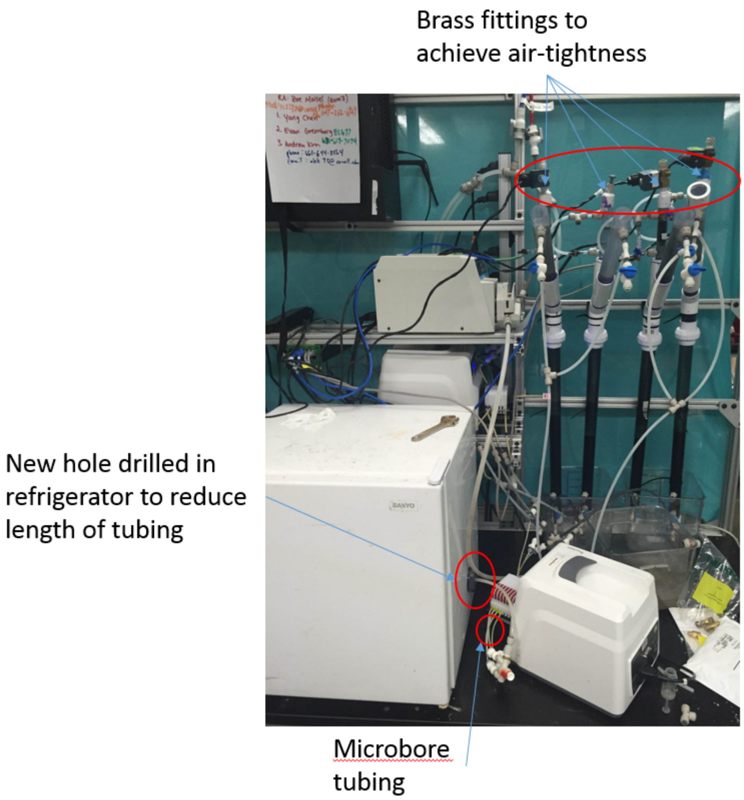

Figure 4: Revised set up with modified refrigeration equipment

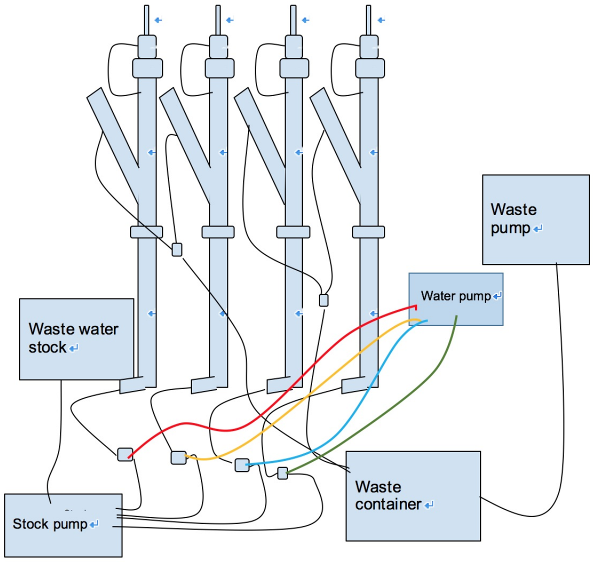

Figure 5: Schematic of revised set up with modified refrigeration equipment

However, simply having shorter influent lines was not enough to cease biomass accumulation in the tubing. Quickly after replacing old influent lines with new ones, biomass accumulation occurred within a couple of days. This can possibly be explained by the fact that connections were not properly autoclaved or cleaned prior to replacing the diluted stock lines. *ZM:* [did you autoclave your connections at all before replacing the stock lines?] Furthermore, not all tubing was replaced when the set-up changed, and the existing biomass contributed to the rapid accumulation. The wastewater stock recipe was also not changed at this time, so non-soluble organic compounds were still settling in the lines.

As a result, to further reduce the clogging in the influent lines, the old quarter-inch diameter tubes were replaced by microbore tubing. The increased velocity through microbore tubing due to the decreased cross-sectional area would reduce the likelihood of particulate matter settling in the lines. However, the issue with using microbore tubing is the likelihood of clogging if the particulate matter is sufficiently large. Microbore tubing was thus only implemented once the stock recipe was modified to reduce the amount of particulate matter. With both the microbore tubing change and modified stock solution, there is little clogging in the lines. This is a promising result that will allow for simpler experimentation and reduced maintenance. Figure 6 shows the new hole on the refrigeration unit, as well as microbore tubing used as influent lines.

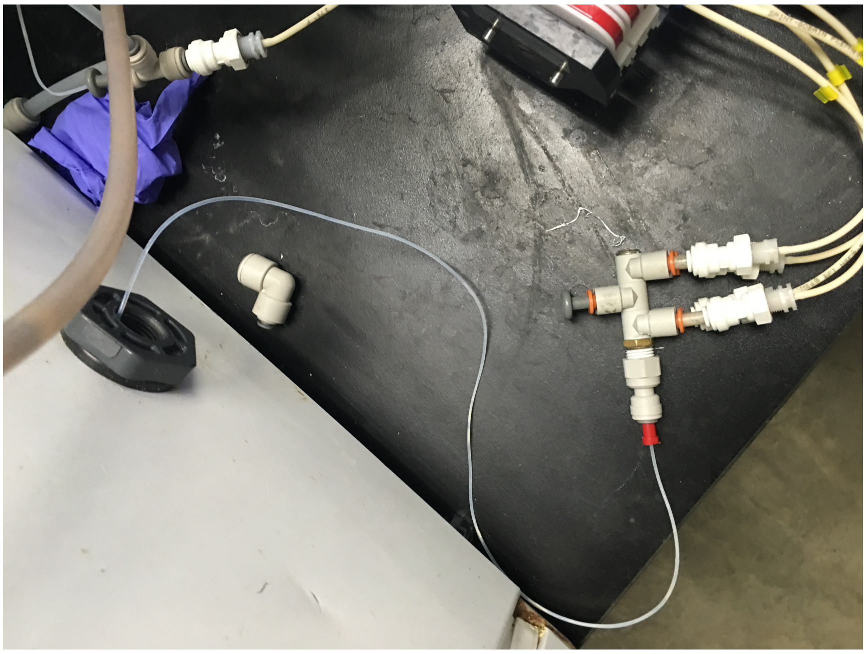

Figure 6: The current set-up of the influent lines

One final modification to the lab set-up was the implementation of ferrule compression brass tubings in the head units of each reactor to establish gas-tight conditions. Push-to-connect parts were found to be not gas-tight during the Summer of 2016. Thus, it was necessary to replace push-to-connects with brass tubing to obtain meaningful biogas collection data. The implementation of ferrule compression brass tubing, as well as non-leaking solenoid valves, have made the reactors gas-tight.

4.2 Reactor R2 Repair

A large crack in the headspace of Reactor 2 (R2) made establishing gas-tight conditions impossible. The large crack in the headspace of Reactor 2 was first sealed using PVC welding wire. Layers of PVC welding wire were placed to cover the crack, but were carefully aligned as to not cover the threaded opening. A threaded unit was screwed on while the welding wire was cooling to mold the wire properly and ensure the threaded hole was still accessible. Figure 7 shows a picture of the repair.

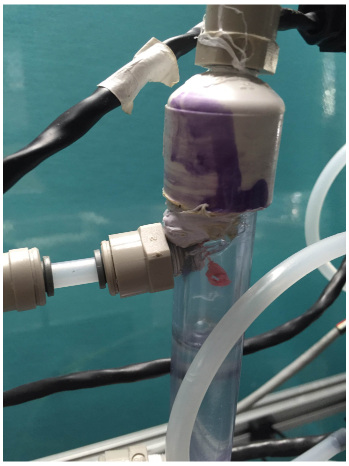

Figure 7: R2 after PVC welding repairs

The PVC welding to repair the R2 headspace proved to be a challenge. It was difficult to effectively seal the leak with welding because the crack was in a very difficult place to operate. As this was also the UASB team's first attempt at welding, a clumsy welding operation occurred. Pressure tests were conducted to determine the success of the PVC welding. It was found that significant leaks still exist in R2. The headspace could not become pressurized at all, which indicated that R2's gas tightness was as non-existent as it was before. It was not completely clear whether this was due to ineffective welding or if another area of leakage exists. However, the success of the ferrule compression brass tubings in the other reactors indicated that the primary cause of leaking was likely due to ineffective welding.

New methods of repairing R2 were explored after the failure of PVC welding. Another option explored was to saw off the top portion of the head space containing the crack, and glue a new cap on. Holes were then drilled and threaded to allow for pressure sensing and connection to the off-gassing valve. The repaired but shortened headspace can be seen in Figure 8.

Figure 8: Repaired R2 with a noticeably smaller headspace than the other reactors

ZM: [check your verb tenses in this section. it switches from past to present tense sometimes at should be primarily in the past tense] Modifying the head unit proved to establish gas-tight conditions in the reactor. However, chopping off a part of the headspace ultimately resulted in less available height for gas to accumulate. After this repair, there was only approximately 1-2 cm between the pressure sensing ports. Although the reactor was gas-tight, this loss in water height was too severe, and the reactor could not effectively off-gas. There were too many off-gassing events to practically analyze the data, and due to the uncertainty in pressure sensor readings, the Procoda file often mistakenly off-gassed even though biogas was not sufficiently accumulated. Details of the off-gassing method are explained in the Methods section below.

Because the off-gassing events in R2 proved to be ineffective due to the decreased available headspace, a new head unit will have to be fabricated. Unfortunately, there was not enough time to fabricate a new reactor, and little justification for doing so when three reactors are running. Therefore, R2 will primarily be used for tests that do not require off-gassing, such as tracer tests to determine hydraulic residence time. Additionally, a bubble meter or methods developed from the Sensor Development team may be implemented for R2 as a method of comparing biogas production from these tools with traditional off-gassing.

4.3 Wastewater Stock Modification

In another attempt to prevent clogging of influent lines, the stock recipe was altered to reduce the formation of chemical precipitates, while still maintaining the integrity of an effective synthetic wastewater solution. A series of tests were conducted to determine which wastewater stock constituents were settling. These include tests for solubility, biological activity, and chemical reactions.

4.3.1 Insoluble Components

Each component of the current wastewater stock recipe (SYNTHES) was tested individually for solubility. The same concentration used in SYNTHES was established in a separate 1 L volumetric flask. Solubility of each component was determined by visual inspection. ZM: [reference the table that shows the SYNTHES ingredients]

Many constituents were found to be soluble in the current wastewater stock recipe. The components in the current recipe were also crosschecked with other existing synthetic wastewater recipes referenced. Many of the current components were deemed necessary and were commonly found in literature, such as ammonium chloride for N, potassium phosphate for P, and calcium chloride for Ca. Fortunately, these constituents were found to be soluble after testing. Some components in the current stock solution were not found in other synthetic wastewater recipes, such as peptone and sodium acetate. These, however, were deemed soluble after testing and were ultimately were kept in the stock solution.

Two main constituents were identified as not soluble. Starch is not soluble at room temperature, and milk powder was found to form large clumps that are reminiscent of the material that settles in the influent lines. Furthermore, these two components were also the largest constituents of the synthetic wastewater stock recipe, with concentrations of around 2 g/L for each. These high concentrations could contribute to the large amount of particulate matter found in the wastewater solution.

Glucose was chosen as a substitute for starch and milk powder due to its solubility, as well as its chemical resemblance to starch. Glucose was also commonly used in other synthetic wastewater recipes examined, with concentrations ranging from 3 to 20 g/L. A concentration of about 4 g/L was ultimately chosen based on these values as well as the concentrations of the components that is was replacing. Glucose was largely soluble and is expected to help with clogging and biomass accumulation problems.

One major area of concern is the loss of hydrolysis rates with these substitution. Starch requires some level of hydrolysation due to the bonds between component glucose units, and milk powder is likely a complex substrate with carbohydrates and lipids. Forgoing these components makes our synthetic wastewater less able to reliably simulate real wastewater. Additionally, by providing more soluble carbon compounds, an increased amount of biogas produced may lead to more plugs forming in the reactors. However, the wastewater stock recipe with these changes is very comparable to other wastewater recipes found in literature, and will be more than sufficient for our purposes in experimentation.

Unfortunately, this recipe still was found to produce particulate matter, which would cause clogging in the influent lines. Figure 9 displays the wastewater stock without starch and milk powder as the primary carbon sources, but still with noticeable amount of particulate matter.

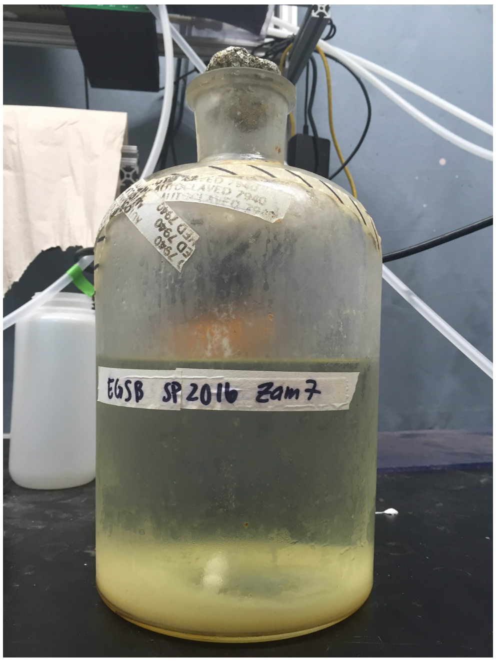

Figure 9: Concentrated stock solution with settled particulate matter

4.3.2 Biological Activity

It was unclear why particulate matter was still forming after implementing soluble constituents. It was thought that the cloudy, particulate matter was a result of biological activity. To test whether this was the case, three experiments were performed: 1) A week-old stock solution was autoclaved, 2) a newly prepared stock solution was autoclaved, and 3) an empty volumetric flask was autoclaved, and stock was immediately prepared in it. If particulate matter formed in any of these experiments, then it can be inferred that biological activity is not the primary cause of particulate matter formation. COD tests were also performed to test whether autoclaving made a significant difference in the organic matter content in the stock.

The results of the three experiments conducted suggest that biological activity is not a factor in particulate matter production. All three groups (old stock autoclaved, fresh stock autoclaved, fresh stock prepared in autoclaved flask) produced settleable matter. The autoclaved flask in which stock solution was immediately made in had the same amount of cloudy particulate matter as the non-autoclaved flasks. Furthermore, settleable matter was found within less than 24 hours in the wastewater solution prepared in a newly autoclaved volumetric flasks, suggesting that the cause is a quick chemical reaction instead of a slow, biological one.

Due to the fact that settleable materials were found in all autoclaved experiments, the cause of particulate matter is likely not a result of biological growth. These results further suggest that there is little need to autoclaving wastewater stock. There are still microbial communities in the wastewater solutions, but it is not likely that there is a drastic reduction in COD from these microorganisms alone.

Additionally, autoclaving the stock resulted in a drastic change in color of the stock solution. It became a dark amber and settled materials were still present in the same quantity as it was before autoclaving. The color change of the stock before autoclaving (yellowish, tan) to after autoclaving (amber, brown) suggests that the composition of the wastetwater stock drastically changes upon autoclaving. Although the COD concentration of the wastewater stock did not change after autoclaving, high heat may be decomposing components in the wastewater solution, which would no longer make the solution an accurate representation of wastewater. The UASB team has decided to not autoclave future iterations of wastewater stock due to these reasons.

4.3.3 Chemical Reactions

To test whether a combination of wastewater constituents were resulting in particulate matter, several experiments were performed. First, all organic compounds in SYNTHES were prepared in a flask, and all inorganic compounds were prepared in a different flask. Observing if settleable material formed in either one of the flasks would allow for further testing. If particulate matter formed in a flask, fewer subsets of the compounds (either organic or inorganic) could be tested in various combinations. In this fashion, the exact components that are reacting could be identified. Table 1 is a list of the components in SYNTHES, with the organic compounds bolded.

The wastewater solution consisting of only organic matter did not show any settleable matter. However, the volumetric flask only consisting of inorganic materials was shown to have particulate matter. As a result, the cause of particulate matter was determined to be a reaction of inorganic compounds. Many different combinations of the inorganic constituents were then tested to see if any particular combinations resulted in reactions. It was found that Magnesium Hydrogen Phosphate ($MgHPO_4$) and Potassium Hydrogen Phosphate (K_2HPO_4), while individually soluble, react to form a precipitate. Furthermore, Ferrous Sulfate ($FeSO_4$) formed a precipitate, which acted as a coagulant and can promote settling of organic compounds.

The ionic components that were found to be reacting, through process of elimination, were magnesium hydrogen phosphate ($MgHPO_4$) and potassium hydrogen phosphate (K_2HPO_4). $MgHPO_4$ was somewhat expected due to the fact that it, while soluble, took a long time to dissolve and often did not dissolve completely. To avoid the precipitates formed from $MgHPO_4$, magnesium sulfate ($MgSO_4$) was used as a substitute. $MgSO_4$ was used in many of the other synthetic wastewater recipes found from the literature review, and using this compound was a natural choice because it is also aqueous.

There are few implications from using $MgSO_4$ instead of $MgHPO_4$. Phosphorus is often the limiting nutrient in microbial growth, and decreasing the amount of phosphorus could slow down the formation of new granules. However, phosphorus is still being provided in the wastewater stock through K_2HPO_4. This component may also be increased in future work if granules are not forming from the lack of phosphorus. Furthermore, the addition of more sulfur from switching to $MgSO_4$ has the potential of decreasing methane production. Sulfate reducing bacteria (SRB) will compete with methanogens for carbon sources. Furthermore, hydrogen sulfide (H_2S) would be produced instead of methane, which further leads to odor problems in the lab. Detection of odor as well as GC measurements may be used to evaluate to limited extent the competition between SRB and methanogenic cultures. However, to truly characterize this competition, genetic testing tool such as qPCR must be used, which is beyond the scope of the team's work.

K_2HPO_4 was found to also form a precipitate when reacting with other compounds, particularly sodium acetate ($NaCH_3COO$). K_2HPO_4 was also an essential compound as it was commonly found in other synthetic wastewater recipes. Furthermore, the reduction of phosphorus from implementing $MgSO_4$ meant that K_2HPO_4 is essential. To address this complication, potassium dihydrogen phosphate (KH_2PO_4) was chosen as a substitute. KH_2PO_4 provides potassium and phosphorus, and this compound was similarly commonly found in other synthetic wastewater recipes. KH_2PO_4 was also found to be aqueous and did not form a precipitate with sodium acetate.

$FeSO_4$ was found to be the primary cause of particulate matter formation. $FeSO_4$ in isolation with water, while initially soluble, eventually forms a precipitate when used in high concentrations over a longer period of time. $FeSO_4$ likely reacted with water to form $Fe(OH)_2$, which is a solid precipitate. This likely had other ramifications, as $Fe(OH)_2$ can often be a coaggulant. In sweep flocculation, organic matter can adsorb to $Fe(OH)_2$ and form cloudy flocs, which results in a reduction of organic matter content. This describes the phenomenon the UASB team saw with the synthetic wastewater, as the particulate matter is often cloudy. $FeSO_4$ was an essential compound in wastewater, so there were no simple substitutes. Instead, the concentration of $FeSO_4$ was reduced from 100 mg/L to 20 mg/L. This value was chosen based on similar values found in other synthetic wastewater recipes. This reduction in concentration is also expected to drastically reduce the settleable organic matter. Although some particulate matter still forms, it was found that with microbore tubing, the amount of settleable material was small enough to not settle in the influent lines. Future teams may consider not including $FeSO_4$ at all if a completely soluble wastewater is desired.

Table 1 below details the newest iteration of the wastewater stock recipe, along with the original SYNTHES recipe for comparison. Further testing is currently being conducted to be absolutely sure that influent lines will not be clogged in future experiments. Fortunately, since the wastewater stock has been modified, there has been less noticeable settling of compounds in the influent tubes. COD tests were conducted for this new wastewater stock solution. The total COD was found to be 7300 mg/L, while the soluble COD was found to be 7000 mg/L. The agreement between total and soluble COD is reasonable due to the fact that the primary goal when creating a new stock recipe was to include more soluble carbon compounds. This value is also reasonable with SYNTHES, which had a total COD value of 8000 mg/L. The N and P concentration have been characterized as much as possible, but the exact formula of some organic components such as peptone and yeast extract are unknown.

current wastewater stock recipe		traditional wastewater stock recipe	
Chemical Constituent	Amount added per liter(mg/L)	Chemical Constituent	Amount added per liter(mg/L)
Urea	1600	Urea	1600
NH4Cl	200	NH4Cl	200
Peptone	300	Na-acetate	1357
MgSO4	395	**Peptone**	300
KH2PO4	305	MgHPO4-3H2O	500
FeSO4.7H2O	20	K2HPO4	305
CACl2-2H2O	120	FeSO4.7H2O	100
Glucose	4100	CaCl2.2H2O	120
Yeast Extract	900	**Starch**	2100
Vegetable oil	500	**Milk Powder**	2000
CuCl2.2H2O	10	**Yeast Ertract**	900
MnSO4.H2O	2	Vegetable oil	500
NiSO4.6H20	5	CuCl2.2H2O	10
ZnCl2	5	MnSO4.H2O	2
		NiSO4.6H20	5
		ZnCl2	5
N	799.0031153	N	799.0031153
P	69.52205882	P	143.4195402

Table 1: Current stock recipe compared with SYNTHES

This new synthetic wastewater recipe is expected to drastically reduce the amount of maintenance requires as well as improve the accuracy of COD readings. With this new wastewater recipe, experimentation was able to proceed smoothly.

5 Methods

5.1 Tracer Experiments

Hydraulic residence time is of interest for characterizing the UASB reactors. Although the volume and flow rate of the reactors were calculated to yield an HRT of 4 hours, it was possible that this value was not accurate. Due to the low volume of the reactors and the relatively large size of the granules, it is possible that there would be preferential flow. Furthermore, characterizing the UASB team's HRT is essential to be able to compare results with the AFB team, who's ultimate goal is to reduce the HRT of anaerobic wastewater treatment reactors.

5.1.1 Red Dye

To determine the true hydraulic residence time of the reactors, a simple dye test was first conducted. The experiment was focused on determining the residence time through the granular bed and not necessarily the time it takes to reach the effluent. This is under the assumption that all degradation of wastewater occurs when it is in contact with granules. To determine this contact time, a pulse of red dye was injected into the reactors while they were operating under normal conditions. Visual inspection was then used to see when red dye first breached through the settled bed of granules.

5.1.2 Fluoride

A fluoride test was also conducted to confirm the results of the red dye test. Using sensitive fluoride sensors instead of visual inspection, these results were expected to provide more accurate data. This test was only conducted on reactor 2 because reactor 2 had a faulty gas collection system, so any negative impacts of fluoride on gas production would not skew the data. A new hole was threaded in R2 right above the granular bed, which served as the effluent port for the test. The fluoride probe was placed right near this threaded hole and continually measured fluoride. Figure 10 shows the set-up of the fluoride tracer test. A pulse of a 50 mg/L solution was injected into the reactor using a pump, and combined with the normal tap water influent line. The fluoride was provided by the chemical company Ricca (CAT Num. 3171-32, 100 ppm). Due to the continuous readings of the fluoride probe, an exit age distribution was constructed.

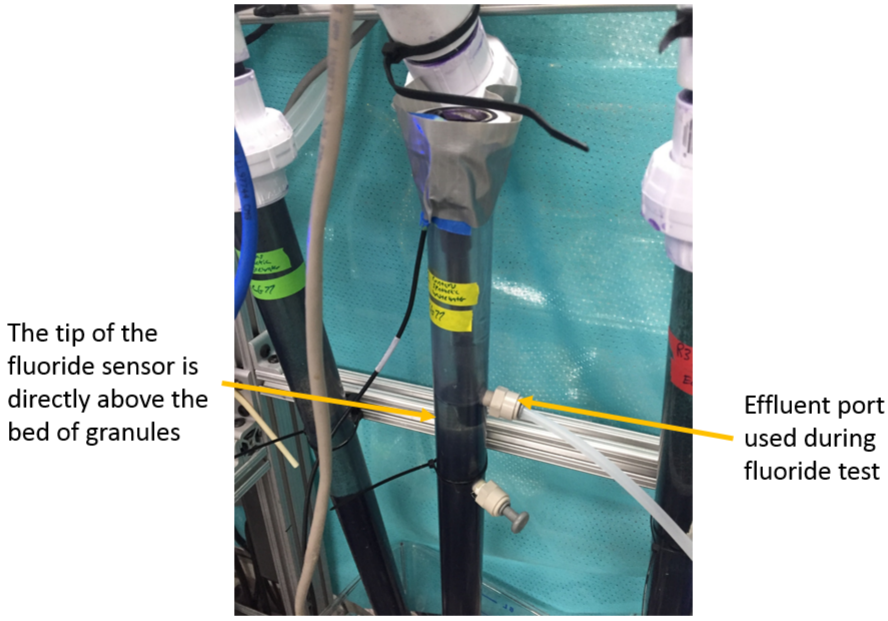

Figure 10: The set-up of R2 during the fluoride tracer test

5.2 Reactor Performance

5.2.1 Biogas Collection and Characterization

Biogas was collected by the same method previous teams have used. A pressure sensor was placed in the bottom of the headspace of the reactor. This pressure sensor detected approximately 4-5 cm of water height above the sensor. As biogas accumulated in the headspace of the reactor, the head space became pressurized and decreased the water height above the pressure sensor. Once the water height reached approximately 2 cm above the pressure sensor, a solenoid valve was opened and released the gas. Once the solenoid valve was opened, the headspace was no longer pressurized, and the water height above the pressure sensor returned to its original elevation of about 4-5 cm. The process of gas accumulation, pressurization, and off-gassing could then be repeated. Through knowing the cross sectional area of the headspace, this method provides the volume of gas produced by the reactors. Figure 8 displays the head units of the reactors and demonstrates this set-up.

The head space above the maximum water height had a rubber septa that allowed for gas chromatography (GC). However, methane concentrations were not well characterized through GC this semester due to lack of sufficient time in experimentation. Because it is well-established that AD produces biogas with around 60-70 percent methane, a theoretical methane percentage of 60 percent is used for subsequent analysis. Thus, through knowing the volume of biogas produced and a theoretical 60 percent methane concentration, the volume of methane can be calculated. This is expressed in Equation 1.

$$V_M = N * H * A * 0.60 \quad (1)$$

Where VM is the volume of methane, N is the number of off-gas events, H is the difference in height of water above the pressure sensor corresponding to each off-gas event, A is the cross-sectional area of the reactor, and 0.6 is the percentage of biogas that is methane.

5.2.2 COD tests

To characterize reactor performance, COD tests were conducted for the influent and effluent. COD tests were performed using pre-made COD testing kits. Two mL of influent and effluent samples were added to the COD vials. After properly mixing and incubating the samples at 150 C for two hours, a spectrophotometer was used to determine the COD concentration. A standard curve for COD developed in the Summer of 2016 was used for analysis. Only one set of COD tests were conducted to characterize reactor performance due to time constraints.

Through knowing the COD concentrations of the influent and effluent, the percent of COD removal can be calculated. This COD removal percentage can also be applied to the mass of COD going in and coming out of the reactors. It is established that using the stoichiometric ratio of methane and oxygen, a theoretical maximum of 350 mL methane/g COD removed can be obtained at standard temperature and pressure. This conversion will be necessary to determine the theoretical amount of methane that should be produced in order to compare with measured methane amounts.

$$COD_R = (COD_i - COD_f)/COD_i \quad (2)$$

The COD percent removal is calculated from Equation 2.

$$V_M = COD_i * Q * COD_R * 350 \quad (3)$$

Equation 3 shows the theoretical methane production based COD removed. Multiplying COD of the influent times the flow rate (Q) yields the mass of COD going into the reactor. The percentage of COD removal and the theoretical 350 mL CH4/g COD removed ratio is then applied.

5.2.3 Reactor Parameters

Table 2 displays the parameters of the reactor that were operated after the set-up, stock recipe, and tracer test tasks were completed.

Summary of Reactor Parameters		
HRT Target	4	hours
Upflow Velocity	0.05	mm/s
Total Flow	1.475	mL/min
Organic Loading Rate Target	8	g COD/L-day
Stock COD Concentration	7300	mg COD/L
Influent COD Concentration	1333.3	mg/L

Table 2: Reactor parameters chosen for operation

An HRT of four hours was chosen due to comparisons in literature. Note that this four hours corresponds to the time through only the vertical part of the reactor. With the given volume of this section known, the corresponding flow rate and upflow velocity were calculated. Details about the exact volume, HRT, and OLR calculations for the reactors can be found in the UASB Spring 2016 report. In the summer of 2016, the reactor was initially inoculated with granules at a volume corresponding to approximately 1/3 of the vertical reactor height. The granular bed eventually naturally increased to about 2/3 of the vertical reactor height as more granules formed. An OLR of 8 g COD/L-day was chosen because it was slightly higher than the OLR operated in the summer of 2016. While OLR was supposed to increase throughout the semester, only an OLR of 8 g COD/L-day was explored and will be characterized in this report.

6 Results

6.1 Tracer Experiments

6.1.1 Red Dye

The red dye test showed that the first sighting of red dye after the bed of granules occurred much sooner than the designed residence time of 4 hours. The time of the first observation of red dye breaching through the settled bed of granules after the pulse input began is tabulated below in Table 3. These quick times warranted further, more sophisticated tracer tests using fluoride.

Reactor	Time for first sighting of red dye (min)
1	25
2	35
3	35
4	25

Table 3: Red dye test results

6.1.2 Fluoride

To characterize the true residence time of the reactors more accurately, a fluoride tracer test was implemented. The results of the fluoride test are graphed in Figure 12.

A standard curve of fluoride test was first developed before starting the tracer test. The concentrations of fluoride were 0, 5, 20 mg/L, which are detected by a probe connecting the sensor listed as Table 4. A plot of the voltage versus log of the concentration was used to develop a linear relationship, as shown in Figure 11.

log(concentration)	concentration of fluoride(mg/L)	volt
0	0	0.06
0.698970004	5	-0.0375
1.301029996	20	-0.0781

Table 4: Fluoride test standard curve table

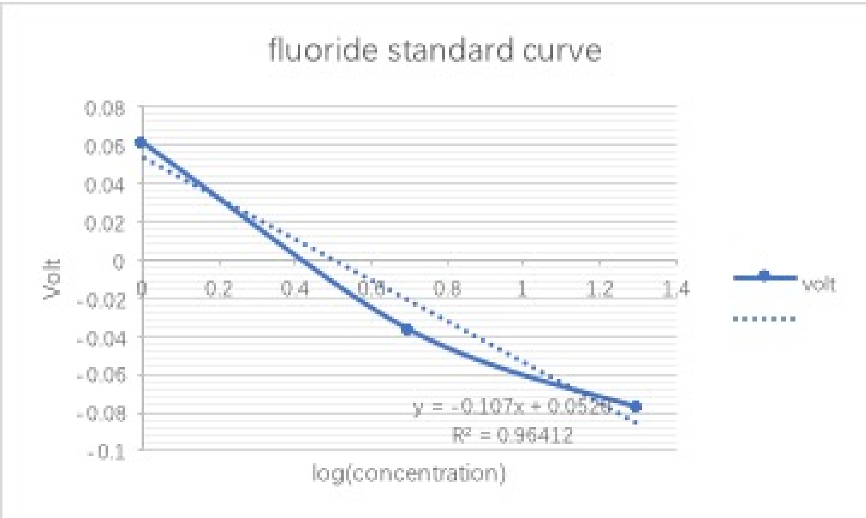

Figure 11: Fluoride test standard curve graph

Using the relationship between the concentration of fluoride and voltage from the standard curve, the results from the tracer test could be analyzed.

The results of the fluoride test in R2 are graphed in Figure 12. The time to achieve the peak concentration is used to estimate the residence time. Thus, the residence time of Reactor 2 is approximately 3.22 hrs.

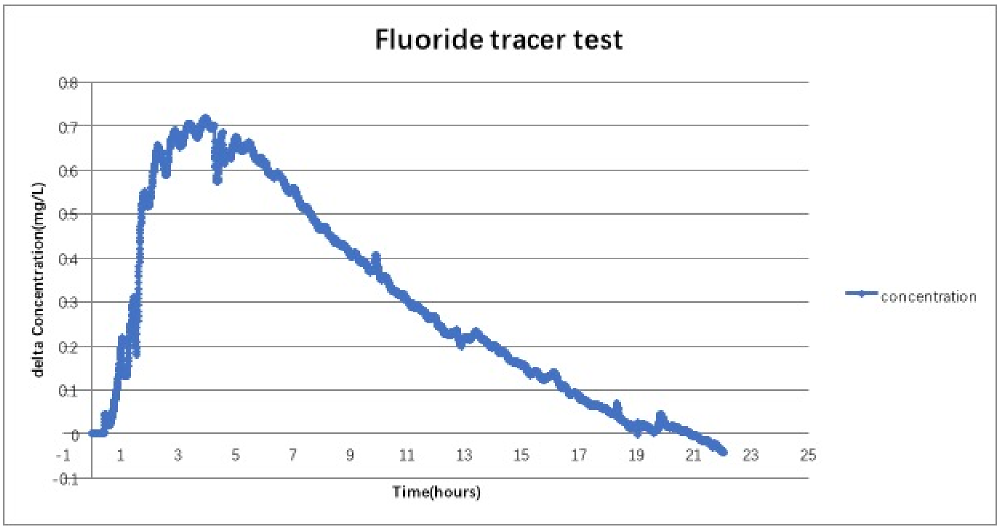

Figure 12: Fluoride tracer test

6.2 Reactor Performance

The UASB team measured reactor performance for an OLR of 8 g/L-day. Figure 13 is an example of biogas collection data under this OLR. Note that only a tenth of a day is shown. Due to the large number of off-gassing events, displaying data for the entire day would be difficult to interpret. Unfortunately, there was not a lot of good data collected for the 8 g/L-day OLR period of testing. This is due to many computer issues and pressure sensor difficulties that were encountered, as well as the limited time available to run this experiment.

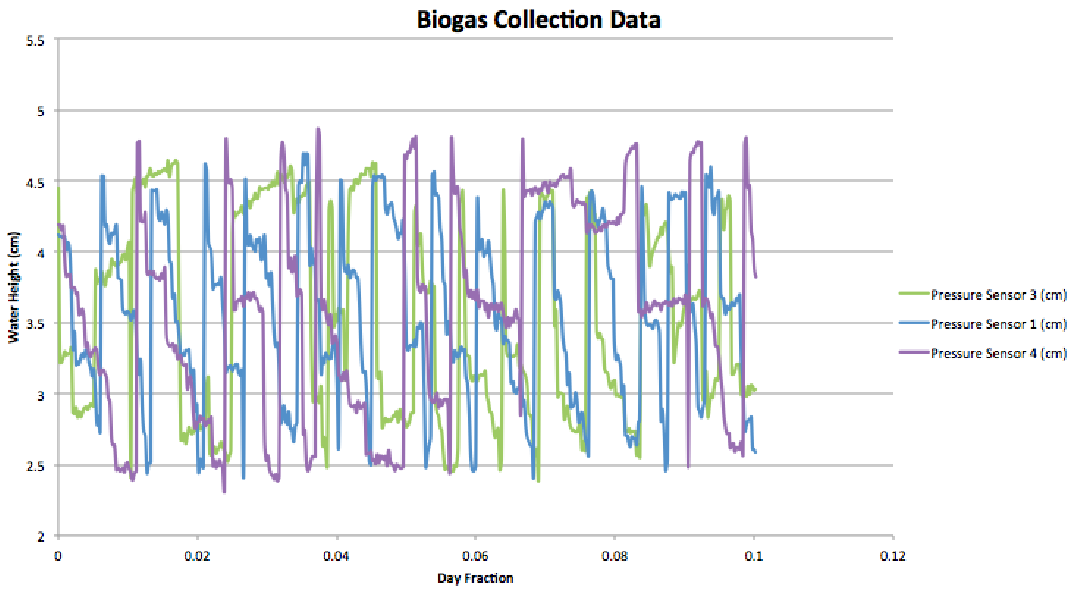

Figure 13: The raw data of biogas collection

COD tests were also quite inconclusive. While the effluent readings indicated very low COD concentrations, the influent readings were much lower than the expected concentration. Influent reading ranged from 100-500 mg/L COD, which is not representative of the theoretical influent concentration based on the known flow rates and COD concentration of the wastewater stock. Table 5 shows the COD removal for Reactors 1, 3, and 4, as well as the corresponding theoretical methane production and actual methane production. R2 data was not characterized due to the inability to collect biogas using the off-gas method.

Reactor	R1	R3	R4
Theoretical Influent COD (mg/L)	1333	1333	1333
Measured Effluent COD (mg/L)	93.45	81.2	105
COD Removal (%)	92.99	93.91	92.12
Theoretical Methane (mL/day)	921.5	930.6	912.9
Measured Biogas Production (mL/day)	1444.1	1119.8	1254.1
% Methane (Estimated)	60	60	60
Actual methane (mL/day)	866.5	671.9	752.4

Table 5: COD results, removal, and theoretical/measured biogas production

7 Analysis and Discussion

7.1 Tracer Experiments

7.1.1 Red Dye

The red dye tracer test revealed a shockingly low residence time. However, this test was simply a preliminary test. The results from the red dye test are likely not characteristic of the true residence time of the reactors. The reactors can be modeled as a plug flow reactor (PFR) with a significant amount of dispersion. The bed of granules, relatively large diameter of the reactor, and high number of expansions caused significant dispersion of the red dye. The time to reach the first sighting of red dye was likely due to these non-ideal conditions, and more representative of the HRT of an idealized PFR (which do not exist) than a PFR with dispersion. Another area of concern with red dye was degradation. Because red dye is ultimately an organic compound, there was some degree of concern of whether or not the granules would degrade the red dye. The level of degradation is currently unknown, but is likely negligible for the purposes of the tracer test.

To characterize the actual residence time with these non-ideal conditions, a method to measure the concentration of red dye would be required. Although a spectrophotometer can do this, the high amounts of solids from cell wash out could potentially damage the spectrophotometer. This analysis was not possible with visual inspection of red dye alone, and thus this prompted the need for fluoride tracer testing.

7.1.2 Flouride

The results from the fluoride tracer test were quite promising. The first instance of fluoride detected by the flouride probe was about 30 minutes, which corresponded to the red dye test. However, from the exit age distribution, it is clear that the residence time is much higher. If the UASB is modeled as a PFR with a significant amount of dispersion, then the residence time would correspond to the peak of the fluoride concentration curve. From visual inspection, the residence time was found to be about 3.22 hours. This is very similar to the target HRT of 4 hours. The discrepancy can be explained from the fact that the calculation of 4 hours was from the volume of the entire vertical portion of the reactor. However, the fluoride probe measured concentrations immediately after the granular bed, which is not necessarily the vertical length of the reactor. In fact, the granular bed was about 2/3-3/4 the height of the vertical section of the reactor, which reinforces the results from the tracer test and the theoretical HRT.

More analysis may be done with the given data. A cumulative age distribution can be determined given the mass of injected fluoride tracer, and the dispersion coefficient can be determined using numeral methods of analysis from the data. Characteristic and mean residence times can also be calculated using numerical methods rather than through visual inspection. These parameters may be explored in the future when the AFB team explores the HRT of the high-rate reactors more closely. However, for the purposes of the tracer test, visual inspection provides a good, promising result.The fluoride test has not been conducted on all four reactors, but each reactor is assumed to be very similar due to the same operating conditions.

7.2 Reactor Performance

A goal for this semester was to determine the maximum organic loading rate of the reactors before reactor failure was reached. Although this maximum OLR was not explored, the current reactor performance reveals valuable information. The results from biogas and COD removal ultimately are promising, but highlight that improvements in measurement methods can be made to obtain more accurate data. Yet, the data collected suggests that the lab-scale UASBs were successful in sustainably treating wastewater.

Although gas-tight conditions for the reactors were established, the off-gassing method posed several complications. First, due to the small size of the reactors, the available water height only ranged from 2-3 cm. This led to very many off-gassing events, and it is expected that the number of off-gassing events will increase with higher OLR. The increased OLR led to a non-continuous flow of biogas with large amounts of biogas being collected at once, which is reflected in the data. Because of the limitations of the off-gassing method, the inconsistent nature of biogas production will lead to difficulty in obtaining readings in the future. Furthermore, the often poor sensitives of the pressure sensor as well as computer issues (random shut offs that led to data not being logged, and necessary re-calibrations of pressure sensors) led to a limited amount of data being collected. Future teams will no doubt benefit from better biogas collection systems currently being developed by the sensor team.

Still, the collected data establishes a rough estimate on the amount of biogas produced. This amount of methane collected is likely to scale up with bigger reactors, and it will be interesting to see how this number compares with the AFB team.

COD test results are also concerning. While the effluent COD concentration was found to be very low, the influent concentration was found to be much lower than expected. Both the stock and water pump were functioning correctly, and the large amount of biogas produced does not seem to suggest that the organic loading rate was lower than expected. The discrepancy between the theoretical and measured influent COD concentration may be due to the lack of mixing in the influent lines. The high velocity through the microbore tubing meant there was not sufficient time for the stock to mix with the tap water line. Because the peristaltic pump for the stock is at a lower RPM than the tap water pump, there is not a steady, continuous stream of stock being delivered. Instead, packets of stock fluid are likely being delivered, which can make sampling inconsistent between each of the reactors. The high variability in the spectrometer readings also gives some uncertainty in COD readings.

Still, when using theoretical COD calculations, the performance of the UASB was in general effective. Ninety percent COD removal is in fact too high to be realistic. This reading may be explained from the soluble wastewater that is being fed, but is more likely a result from inconsistent COD data. Making sure COD results, as well as increased frequency in COD testing, will be necessary for future semesters.

In general, effluent from the reactor was low in COD, and biogas collection was partially characterized. An OLR of 8 g COD/L-day seemed to be an effective amount of organics input for the reactors based on these results.

8 Conclusion

Over the course of the semester, various changes have been made with differing level of effectiveness. The new set-up with shorter and smaller diameter influent lines proved to be a welcome change. The synthetic wastewater recipe change includes substitutions of glucose for insoluble carbon compounds as well as substitutions for compounds that form chemical precipitates. This new recipe is promising for its ability to reduce settling of organic compounds in influent lines. The fluoride tracer test revealed a detailed profile of transport in the reactor, which will be helpful for future work in the AFB team. The reactor performance at the given OLR is promising but currently limited, and the characterization of UASB reactors is expected to continue with confidence as experiments increasing the OLR continue.

9 Future Work

Future testing will still be needed to determine the maximum organic loading rate the lab scale reactors can handle before failure. Slowly increasing the OLR over the course of a semester will be a necessary task future teams can perform to characterize maximum OLR. Future work can also focus on the the newly discovered hydraulic residence time. Experiments to increase effective hydraulic residence time by adding more granules or fluidizing the previously settled bed of granules could be potential avenues to increase efficiency of the UASB reactors. Additional tasks future teams could explore include utilizing AguaClara drinking water treatment technology for wastewater treatment. Pairing an Aguaclara sedimentation tank with the UASB reactors could potentially increase treatment efficiency and reduce turbidity and pathogens discharged into the environment. This also has the potential to reduce the amount of chlorine required to reduce the pathogen level of the effluent to an acceptable amount. The engineers in Honduras have constructed a 1 L/s UASB reactor that the students travels to Honduras in January will see. This reactor opens doors for experimentation in Honduras and also indicates a genuine interest in and dedication to wastewater treatment in Honduras. Building on the work completed this semester, the outlook for Aguaclara Wastewater teams looks very bright.

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Semester Schedule

9.1 Task Map

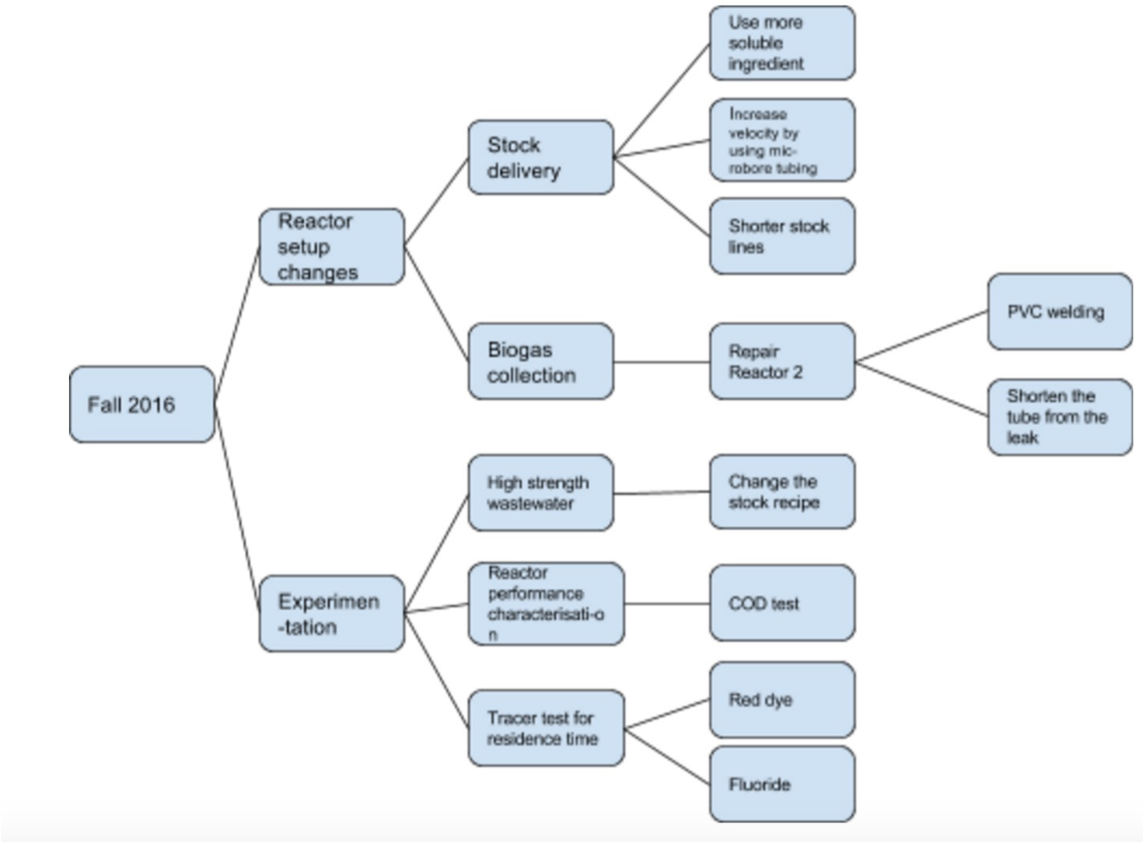

Figure 14: Task map for UASB team Fall 2016

Task List

1. Reactor Setup Changes

   (a) Stock Delivery

      i. Biomass accumulation in wastewater influent lines have shown to be a large problem. Influent COD measurements are larger than what they theoretically should be due to the biomass. This also calls for additional unnecessary maintenance that can be avoided.

      ii. One possible suggestion (by Monroe) is to use microbore tubing to create an increased velocity in the influent lines. Any biomass will not be able to settle. However, this may not be feasible due to clogging of the microbore tubing.

iii. Alternatively, modify the stock recipe to include more soluble compounds. Other papers that include a synthetic wastewater recipe will be examined, and a new wastewater stock may be developed.

iv. Look into other synthetic wastewater recipes, and coordinate with AFB. Andrew Kim has already compiled a potential list of wastewater stock recipes from his past work.

(b) Repair Reactor 2

i. Large crack in R2 headspace makes biogas collection impossible with the current system.

ii. One potential solution to remedy this crack is to weld PVC to possibly repair the leak.

iii. We will fabricate a new head unit if necessary.

(c) Biogas Collection

i. Off-gassing events is not a sustainable solution due to large number of events as well as biomass accumulation in headspace. We will attempt to make the off-gas collection system as accurate as possible for experimentation until a better system is developed.

ii. Replace push-to-connect tubings with ferrule compression tubings to further establish gas-tightness. A ferrule fitting has shown to be very effective in R1 to establish gas tight fittings. Three more will be ordered for the other reactors. Brass hex-reducing nipples, which screw into the cap on the top of the headspace, will be ordered for each reactor (4) to replace the plastic ones to further prevent gas leaks.

iii. Look into methods of biogas collection independent from headspace of reactor. This may include using a gas meter or an external biogas collection chamber.

iv. Collaborate with Sensor team on getting methane sensors working for future use.

2. Experimentation

(a) High-Strength wastewater

i. Feed "black water" / high strength wastewater to reactors.

ii. Change the stock recipe to increase organic content and better simulate black water. Influent concentrations of 10 g/L may be sufficiently considered as "blackwater" (Graaf 2010).

iii. Increase organic loading rate and hydraulic retention time to maximize production of biogas, until reactor failure is reached. OLRs reported in papers treating black water will be the target (1 kgCOD/m3-day), but reactor failure is predicted to occur before this target. Maximum HRTs typically range around 4 hours due to reactor constraints, and any variation of residence time will not differ signficantly. (1 hour HRTS were tested in the summer and proved to be largely ineffective)

iv. Be wary of conditions that lead to reactor failure due to hyperaccumulation of volatile fatty acids. pH tests can be an indication of acidification, but this method is poor because it occurs after the fact. We will look into ways to measure VFAs, either total VFAs through standard assays or by measure individual VFA concentrations through GC or HPLC (neither of which are readily accessible in Hollister Hall).

(b) Tracer Tests

i. Determine mean hydraulic residence time to better manipulate reactor parameters and to assess extent of short circuiting or dead space in reactors.

ii. Begin with red dye for simplicity.

iii. If red dye is ineffective due to organic properties, look into conservative tracers (fluoride, bromide).

iv. Determine the extent to which reactor tilt angle affects residence time. The reactors are currently at an angled tilt in order to establish an appropriate free surface in the head space. Assessing the extent to which these angles affect the residence time will be interesting to see. Ideally once methane sensors are developed reactors will be completely up-right.

(c) Reactor Performance and Characterization

i. COD tests of influent and effluent to characterize COD removal. COD tests will be run at least once a week once experimentation occurs. One average week of COD measurements will likely contain about 20 samples (4 reactors, influent and effluent, stock concentrations, blanks, and duplicates). Frequent COD measurements will be costly so ordering materials to prepare own reagents may be something to look into.

ii. GC for methane concentration measurements, using the GC in the teaching lab, will also be conducted at least once a week.

iii. pH tests using pH paper will be conducted occasionally until pH probes are ordered.

iv. Granule characterization will possibly be conducted. Ports are in the reactors to potentially draw samples from the sludge bed. They will be measured for physical size and shape. Biological characterizations (specific methanogenic activity, biological methane potential, qPCR) are beyond the scope for this semester.

v. Look into possibility of measuring other important parameters such as TKN, TAN, VFAs.

vi. Daily maintenance such as cleaning delivery lines and reactors, replacing stock, leak checking.

3. Analysis

(a) Modeling

i. Use data from UASB experiments to model applications in Honduras based on previous work from Erica Gardner.

(b) Writing

i. Analyze and interpret results for final written report. Compare results with past teams..

Team Coordinator: Andrew Kim

Materials Coordinator: Evan Greenberg