Fluoride Floc Blanket, Spring 2017

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Abstract

The Fluoride subteam seeks to develop a sustainable, inexpensive fluoride removal system for implementation in upcoming AguaClara plants located in India. After earning an EPA Phase II grant for the Spring 2016 fluoride removal reactor, the subteam seeks to improve fluoride purification by testing lab-scale systems to compare a single reactor with reactors in series. At the beginning of this semester, the subteam identified potential issues with floc buildup at the bottom of the reactor. Thus, a sloped plane bottom geometry was incorporated into the reactor system to encourage recirculation of the flocs. Additionally, experiments with high concentrations of PACl resulted in clogging of the apparatus due to PACl buildup. Clay was incorporated into the influent stream to abate this PACl buildup. Initial testing of fluoride removal with the updated one and two reactor systems provided results that seem to indicate slightly better fluoride removal efficiency with two reactors, but more data collected by future testing is required to make a concrete conclusion.

Introduction

With 85% of its drinking water sourced from groundwater, India is the largest user of groundwater in the world. In stark contrast to tap water sources in the United States that are supplemented with fluoride, India's groundwater sources often display excess levels of fluoride, making villagers who rely on these well sources for water at a high risk of overexposure to fluoride. The prevalence of dental fluorosis, an indicator of excessive fluoride concentrations, differs across India, but has been shown to range from 13-91 percent depending on the age group in question and the water source supplying the state or municipality (Arlappa et al., 2013)

In accordance with AguaClara's mission to create affordable, reliable, and sustainable water treatment solutions, the goal of the subteam is to create such a solution for groundwater with excessive fluoride concentrations. Toward this goal, teams from previous semesters analyzed the efficiency of fluoride removal by passing a coagulant, polyaluminum chloride (PACl), and a solution of fluoride through a sand filter. However, the sand filter was an inefficient method of removal because of the buildup of headloss from particles deposited in the sand filter that eventually prevented water from flowing through (Dao et al., 2015). Thus, instead of using a traditional sand filter, the team researched a similar relationship between PACl and fluoride via a floc blanket reactor. In the floc blanket reactor, flocs of PACl and clay adsorbed the fluoride from the influent water. The flocs then overflowed into a floc weir as the floc blanket grew and the purified water flowed out of the top of the reactor. This reactor was modeled after the floc blanket, floc weir, and plate settlers setup in the sedimentation tank of a typical AguaClara water treatment plant. The team expected that the floc blanket reactor would be able to remove fluoride with a significantly higher efficiency than the sand filter from previous semesters and could run for extended periods of time due to the absence of headloss buildup in the reactor. The flocs in the floc blanket could exit the floc weir while the fluoride in the sand filter would just build up and saturate the sand in a short amount of time (Longo et al., 2016) (Cheng et al., 2016). This semester, the subteam continued the work that was started last semester comparing the removal of dissolved species, particularly arsenic and fluoride, from groundwater between two reactors in series and a one reactor system. Red dye was used in place of fluoride for the majority of testing since red dye is cheaper and easier to see in the reactor and red dye and fluoride have similar properties in how they are removed and adsorbed to PACl. Once satisfactory removal efficiency was achieved, the team switched to using fluoride to determine more accurate conclusions. Theoretically, two reactors in series will allow the flocs to interact with and remove more red dye than a single reactor could, therefore using the flocs to their full potential and resulting in overall cleaner water. The reactors will also be operated at concentrations of PACl significantly higher than that of red dye to mimic conditions in the field.

Literature Review

Fluoride Limitations and Hazards

Over-consumption of fluoride can lead to arthritis, dental fluorosis, crippling fluorosis, bone deformation and ligament calcification (Roholm, 1937). Fluoride can cause irritation through inhalation, digestion, and touch and can cause damage to both eyes and exposed skin (NJ Department of Health, 2010). Though there isn't an established "average" level of fluoride in India, the literature suggests that fluoride levels are seldom above 5 $\frac{\text{mg}}{\text{L}}$ in groundwater. However, in the remote Karbi Anglong district of India, fluoride levels range from 5-23 $\frac{\text{mg}}{\text{L}}$ causing severe anemia, stiff joints, painful and restricted movement, mottled teeth and kidney failure (LeChevallier and Au, 2004).

According to the National Research Council (NRC), the maximum contaminant level (MCL) of fluoride in drinking water is 4 $\frac{\text{mg}}{\text{L}}$. However, a secondary limit of 2 $\frac{\text{mg}}{\text{L}}$ has been established by the EPA to avoid potential cosmetic effects such as tooth and skin discoloration. The World Health Organization (WHO) established a safe upper limit of 1.5 $\frac{\text{mg}}{\text{L}}$ to avoid all potential risks of fluoride consumption. The team will be striving towards the WHO guideline of 1.5 $\frac{\text{mg}}{\text{L}}$ of fluoride this semester by designing and experimenting with the floc blanket reactor and manipulating the ratios and concentrations of PACl and clay in the system.

Polyaluminum Chloride (PACl) and Fluoride Removal

One common type of water treatment consists of a series of coagulation, flocculation, and clarification. During coagulation, raw water is mixed with a positively charged coagulant (typically an aluminum salt or iron salt), altering or destabilizing any negatively charged particles or dissolved and colloidal contaminants (EPA, 2016). Depending on the dose of coagulant, there are two methods of particle destabilization. The first, charge neutralization, occurs with a lower coagulant dose and happens as the negative colloids are attracted to the positively charged coagulant particles. The second method, sweep flocculation, requires a very high coagulant dose and transpires when the contaminants are caught by precipitates as they settle in the suspension (EPA, 2016). The destabilized particles then proceed through flocculation, where additional mixing increases the rate of particle collision, forming larger precipitates. Following the formation of flocs, clarification removes the agglomerated particles through sedimentation or other removal processes (EPA, 2016).

In recent years, polymerized forms of aluminum salts have been used increasingly to replace standard aluminum salt coagulants (Ingallinella and Pacini, 2001). Polyaluminum chloride, a partially hydrolyzed aluminum salt is one of the most widely used, as it delivers results similar to aluminum sulfate coupled with a polyelectrolyte (Ingallinella and Pacini, 2001). The main advantages of using polyaluminum chloride instead of alum include a reduction in sulfates added to treated water, lower sludge production, reduced odor problems, and overall higher removal efficiency (Gebbie, 2001). In the Daylesford Water Filtration Plant, a dose of 45 $\frac{\text{mg}}{\text{L}}$ of alum was required to produce potable water, while only 12 $\frac{\text{mg}}{\text{L}}$ of PACl. Additionally, PACl is advantageous in particulate removal because its hydrolyzed state allows for it to be less affected than typical aluminum salts when temperature conditions are inconsistent (EPA, 2016). Furthermore, PACl has a broader range of raw water pH in which it is an effective coagulant. It shows stable turbidity removal from 5.0-8.0 pH, compared to a range of 6.0-7.0 pH for both $AlCl_3$ and $Al_2(SO_4)_3$ (Yang et al., 2010). For the removal of fluoride, PACl has been found to be the most effective with pH values between 5.2 and 6.2 (EPA, 2016).

Several techniques are currently in existence that use PACl to specifically reduce high fluoride levels. The Nalgonda technique is a popular fluoride removal method that involves a combination of rapid mixing, flocculation, sedimentation, filtration and disinfection. However, fluoride removal is usually done through co-precipitation (Bailey and Fawell, 2004). The technique has traditionally been done using aluminum sulfate, but more recent experiments have proven that PACl can be an effective substitute (Kumbhar and Salkar, 2014). The Nalgonda technique typically utilizes a "batch filtration" method, where large quantities of water are treated in buckets. This technique does not utilize continuous flow, and requires a series of treatments to obtain decontaminated water for extended periods of time. For this reason, the Nalgonda technique has been largely introduced as a household treatment method, and has been introduced to various Indian villages, including those in Nalgonda and in the state of Telangana. It is also currently being studied at the pilot scale in Kenya, Senegal and Tanzania (Dahi et al., 1996). In addition to the restrictions implied by batch treatment, the Nalgonda method requires a high dosage of aluminum sulfate to aggregate with fluoride and precipitate. A study conducted by (Dahi et al., 1996) suggests that 13 $\frac{\text{g}}{\text{L}}$ alum (1.2 $\frac{\text{g}}{\text{L}}$ as Al) is needed for the Nalgonda method to effectively treat fluoride levels between 9 and 13 $\frac{\text{mg}}{\text{L}}$. Despite the high concentrations of coagulant, the fluoride residual in the test was still unable to meet the WHO safety guidelines of 1.5 $\frac{\text{mg}}{\text{L}}$ of fluoride. The high dose of aluminum sulfate also leaves high sulfate residuals in the water, which causes taste and odor issues (Bailey and Fawell, 2004).

In regards to other filtration methods, a study by Inganiella achieved 33.3% removal of fluoride using a combination of a gravel pre-filter and a sand rapid filter to capture granules of fluoride, PACl, NaClO and SO_4H_2 (Ingallinella and Pacini, 2001).

Floc Blankets

Floc blankets develop when vertical flow sedimentation tanks form a fluidized bed of particles that then facilitates particle removal by "increasing particle-particle interactions that lead to flocculation and filtration occurring in the floc blanket" (Hurst, 2010). The process of forming flocs requires both the precipitation of aluminum hydroxide from the coagulant and contact with raw water colloidal particles (Hurst, 2010). Once the combination of precipitation and mixing forms small particles, those new flocs collide to form larger, more porous flocs that can then be used for clarification (Hurst, 2010).

This floc blanket clarification is considered hindered settling, which is a form of sedimentation (Gregory et al., 1996). Sedimentation processes are characterized by the removal of suspended particles, e.g. flocs, sand and clay, from water. Removal is possible due to the differences in density between water and the suspended particles, but is also dependent on the size of the suspended particles, water temperature, turbulence, stability of flow, bottom scour and flocculation (Sun, 2004). Floc blanket clarification, however, is primarily governed by upflow velocity of the water and by floc concentration (Gregory et al., 1996). The relationship between upflow velocity, concentration and water quality can be combined into the mass rate of settling, which is equal to the product of the upflow velocity and concentration. Within a range of mass fluxes, a distinct interface is established between clear water and the floc blanket and thus appropriate values of velocity and floc concentration can be deduced. At concentrations above that ideal mass flux range, the aggregation of flocs becomes thick enough that compression settling occurs. At concentrations below that appropriate range, flocs are not inhibited by other particles and a suspension with different settling velocities is formed (Gregory et al., 1996).

Floc blanket clarification is used to purify water in many ways around the world. In Taiwan, a process of pre-sedimentation, floc blanket clarification and sand filtration is used to reduce 100 NTU water down to potable levels (Lin et al., 2004). Floc blankets have also been used extensively to purify water of algae, protozoa and specific virus strains (LeChevallier and Au, 2004). Therefore, it is believed that the adaptability of this method in conjunction with the use of PACl will allow for effective fluoride removal.

Coagulation

Many sources have shown that PACl seems to be the optimal coagulant for the purposes of the project which are to remove arsenic and fluoride from the water being treated (Zonoozi et al., 2009). PACl has been and continues to be used as the most common form of dye removal at the industrial scale. A main reason as to why PACl is so popular as opposed to other coagulants despite its cost is that PACl does not cause any leeching of hazardous chemicals into the water from dye decomposition (Golob et al., 2005). A drawback of PACl is that once its coagulation capabilities are saturated, the dye coagulation drops drastically while other coagulants are able to reach a maximum capacity and plateau. The team has been using PACl as the coagulant since the start of the project, thus for the sake of consistency in results, PACl will be used until the reactor design can be optimized. Only once the reactor is optimized can coagulant experimentation be considered.

Countercurrent Flow Theory

The use of countercurrent flow for separation of clean and dirty water is a major component of the Countercurrent Stacked Floc Blanket Reactor (CSFBR) system. Countercurrent flow is a common concept that can be observed in biological systems such as the kidneys. Countercurrent parallel vessels exchange NaCl through passive diffusion to remove excess NaCl from fluid that would then be circulated throughout the body (Kokko and Rector, 1972). The CSFBR utilizes the concept of countercurrent flow to decrease the concentration of dissolved species in reactor 1 by moving flocs from an area of low concentration of dissolved species (reactor 2) to a high concentration of dissolved species (reactor 1). This is accomplished by removing the flocs from reactor 2 using the floc hopper and directing them to the influent stream of reactor 1. Consequently, the effluent flocs from reactor 1 will be more concentrated with the dissolved species and the system will produce less waste per mass of contaminant removed (see Figure 3.

Previous Work

In the spring of 2016, the team analyzed data that suggested that the sand filter system was inefficient and decided to move towards the idea of a single floc blanket reactor (to see details about this system, refer to Longo et al. (2016). Although the sand filter provided cheaper, adequate removal of fluoride per milligram of PACl used, a key issue that arose with the sand filter was the system run time. The sand filter became saturated with PACl and fluoride too quickly and the head loss built up rapidly. In a matter of a couple of hours, the floc blanket was completely saturated to the point where it was no longer efficient or providing adequate removal of fluoride. Consequently, the system had to be backwashed too frequently to be an effective process. On a much larger scale, in a full-sized plant for example, such a reduction in time to failure would require more maintenance than is feasible. In order to address this, the team fabricated a new reactor mirroring that of the floc blanket and plate settlers in the current AguaClara plants. The team set up a new apparatus fit with stock tanks, a reactor, a turbidimeter, a flocculator, and stock and waste pumps, referencing research previously conducted on the relationship between the amount of coagulant added and head loss accumulation (Dao et al., 2015). The team then developed a MathCad file to calculate flow rates of pumps from a given set of parameters including upflow velocity, tubing diameters, and reactor concentrations. The team also created a ProCoDA method file to turn the flow rates into RPMs for the pumps so that the process of changing PACl and fluoride concentrations within the reactor was more user-friendly.

After the calculations and fabrication were completed, the team was able to successfully create a floc blanket composed of clay and PACl. When the floc blanket had stabilized, the team then tested the system with fluoride. By performing short term tests that last about 10 hours, the team was able to remove around 85% of the initial concentration of fluoride. The final concentration of fluoride in the effluent was lower than the WHO regulation, indicating that the floc blanket reactor was a viable method to remove fluoride (Longo et al., 2016).

In an effort to improve reactor efficiency, the team fabricated an entirely transparent version of the reactor, where the important contact points (the floc weir and the bend to the tube settler) were visible so that interactions occurring in those areas could be analyzed. The team then used this reactor at an EPA competition to analyze dye in place of fluoride and found that this was a successful dye removal apparatus. Finally, the team performed some initial jar tests which suggested that the use of clay may not be necessary for the removal of fluoride (Longo et al., 2016). If it were experimentally verified, then the elimination of clay from the system design could reduce the operating cost needed for fluoride removal.

In the fall of 2016, the team fabricated a new bottom insert to prevent the accumulation of flocs which clogged the bottom of the reactor in the past. The newly fabricated bottom geometry with a smooth sloped bottom allowed for gradual flow expansion and recirculation of flocs that would have settled to the bottom of the reactor with the old bottom geometry. Various concentrations of dye were tested (from 25 $\frac{mg}{L}$ to 100 $\frac{mg}{L}$) using a 1:1 PACl to dye ratio to see whether higher concentrations of dye would clog the reactor. However, even though increasing the concentration of dye increased the density of the floc blanket, the reactor did not clog and the bottom insert proved to be successful for these tests (Cheng et al., 2016).

The team also set out to determine the minimum length of the reactor needed to save resources and space. A shorter reactor, with a height of 5 cm below the weir was tested with concentrations of 25 $\frac{mg}{L}$, 50 $\frac{mg}{L}$, and 100 $\frac{mg}{L}$ of dye. Although the floc blankets reached a steady state height of around 20-30 cm in the short term, the reactor failed as flocs built up and went up through the tube settler to the turbidimeter in the long term. This suggested that there is a minimum height that a floc blanket will reach and the shorter reactor system is therefore not feasible (Cheng et al., 2016).

During the final portion of the semester, the subteam performed long duration experiments with the long reactor in order to compare its results with those of the CSFBR subteam. The CSFBR subteam had hypothesized that a species removal system composed of two reactors in series could lead to cleaner water than the Fluoride team's single reactor system. During each iteration, the two teams ran each system for approximately twenty four hours with the same red dye and PACl concentrations. However, the results of these experiments were inconclusive because the CSFBR team found that the PACl would clog their system midway through each experiment, rendering the reactors incapable of further purification (Dokko and Espada Fraile, 2016). Thus, the current subteam seeks to reexamine the benefits of reactors in series versus a single reactor and conclude which system yields the lowest effluent concentrations of red dye and fluoride.

Methods

Experimental Apparatus

The construction of this apparatus and the fabrication of the reactor was completed last semester. The list below references materials used in the making of the entire apparatus and Table 1 lists the necessary constraints on the reactor for it to perform as designed. To view complete instructions for apparatus construction and a more in-depth discussion of design and calculations, please refer to the spring 2016 AguaClara report (Longo et al., 2016). For information on how to run an experiment, refer to the manual at the end of this report.

The reactor system was setup in such a way that it had the ability to be used as either a one reactor system or a two reactor system by turning several valves. The effective setup of the one-reactor system can be seen in Figure 1 and the effective setup of the two-reactor system can be seen in Figure 2. When running a one-reactor test, Reactor 2 (on the right in the two-reactor system) is used. The entire apparatus at the lab bench is shown in Figure 3.

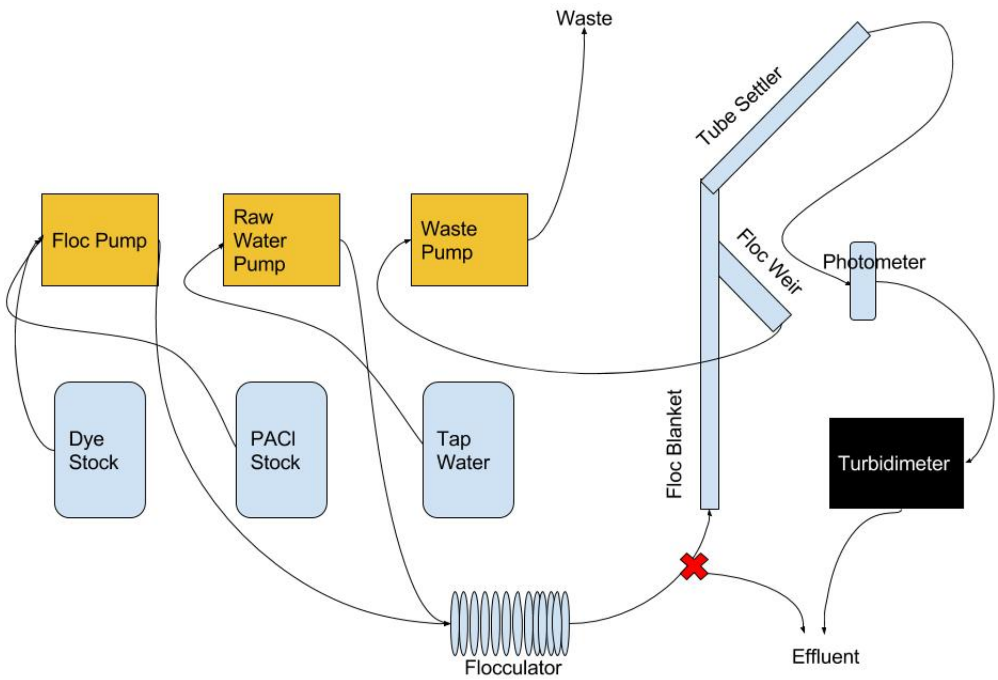

Figure 1: One reactor schematic

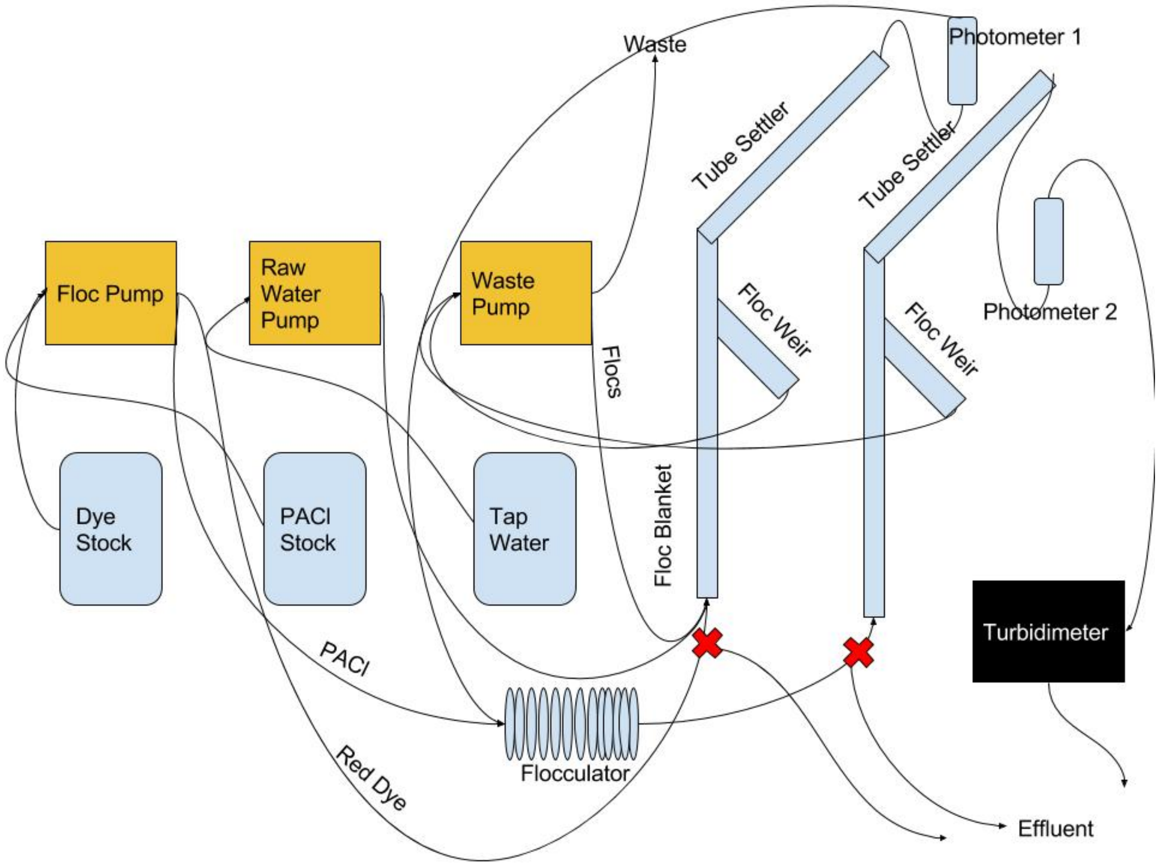

Figure 2: Two reactor schematic

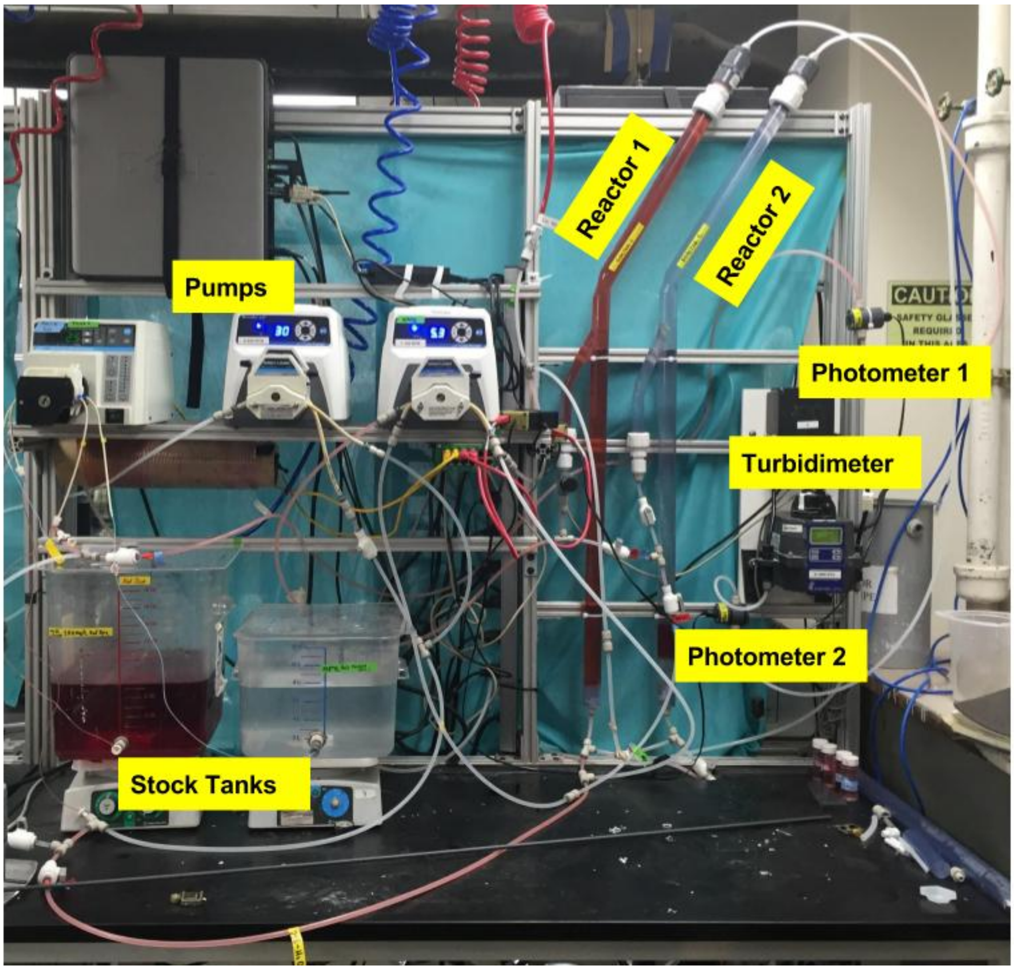

Figure 3: Picture of the full apparatus setup

Water Flow Through Reactor

1. Water and red dye mixes together and then combines with flocs from the waste of reactor 2 and enters reactor 1.
2. The red dye, water, floc mixture then forms a floc blanket where the flocs go over the weir and the cleaner water goes out the top of the reactor into photometer 1.
3. The water then flows through photometer 1 and mixes with PACl.
4. The PACl and mixture from the effluent of reactor 1 then goes through the flocculator and enters reactor 2.
5. The red dye, water, and PACl mixture makes flocs that travel through the reactor, forming a floc blanket. The flocs fall through the weir and the clean water goes out the top of the reactor.
6. The effluent of reactor 2 then goes through photometer 2 and through the turbidimeter into the waste line.

This system of water flowing through the reactor is what represents the counter current flow theory. The dirtiest water will be met with the dirtiest flocs from reactor 2 where some removal will occur. Then the water with less contaminant will mix with pure PACl to provide the necessary removal to the appropriate levels of contaminant for safe drinking. (see Figure 4) (Dokko and Espada Fraile, 2016).

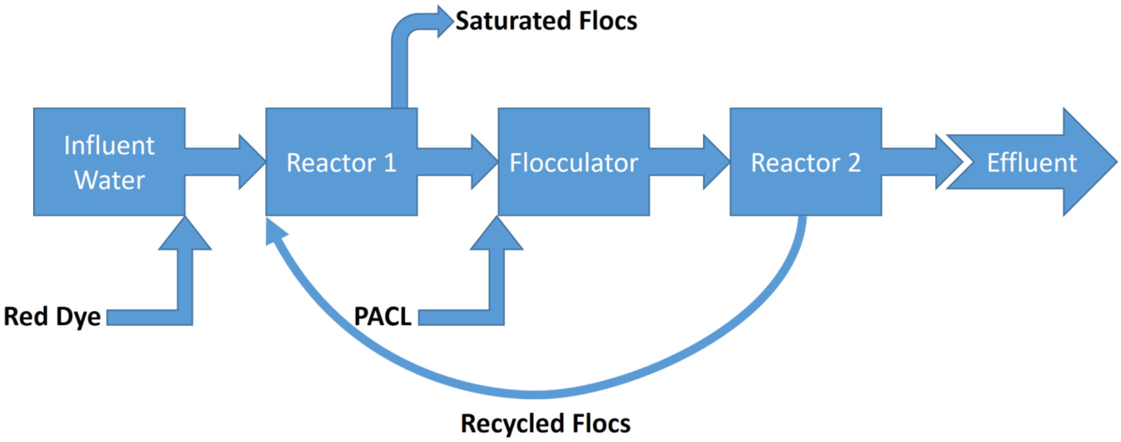

Figure 4: Water flow through the 2-reactor system

Materials

- Two 600 RPM pumps and one 100 RPM pump
- Transparent 2.54 cm (1") PVC piping
- Flexible and hard 0.635 cm (1/4") tubing and Microtubing
- Turbidimeter
- Polyaluminum Chloride (PACl), Red Dye #40
- Various connectors and buckets for stocks
- Two stir plates with stir bars
- 2 Photometers
- Push-to-Connects and Valves

Table 1: List of important parameters for reactor

Parameter	Symbol	Value
Residence Time	θ	4513 s
Hydraulic Gradient	G	75.2 s^{-1}
Velocity Gradient	$G\theta$	13000
Upflow Velocity	v	1 mm/s
Capture Velocity	v_{cap}	0.12 mm/s
Total Flow Rate	Q	30 mL/min
Floc Flow Rate Percentage	Q_{Floc}	20%
Water Flow Rate Percentage	Q_{Water}	80%
Reactor Floc Blanket Length	L	24 inches
Flocculator Tubing Length	l	46 feet
Tube Settler Length	L_{TS}	14.75 inches

[Refer to the manual for detailed information about how each test was run].

Iterations

0.1 Bottom Geometry Testing

0.1.1 Procedure

In the fall of 2016, the CSFBR team had sludge buildup in the reactor running at 1 $\frac{mm}{s}$ (Dokko and Espada Fraile, 2016), so the Fluoride team this semester tested the same apparatus to see if there was continued sludge buildup. The team tested two concentrations of dye (5 $\frac{\mathrm{mg}}{\mathrm{L}}$ and 25 $\frac{\mathrm{mg}}{\mathrm{L}}$) with the same concentration of PACl for each test (25 $\frac{\mathrm{mg}}{\mathrm{L}}$) resulting in tests with different dye to PACl ratios to see if higher PACl to dye ratios would result in PACl sludge buildup. After running these tests, the team found that sludge built up at the bottom of the reactor as seen in Figure 6. After examining the bottom geometry of the second reactor, the team discovered that the CSFBR team used a $\frac{1}{4}$" jet while the Fluoride team used a $\frac{1}{8}$" jet as seen in Figure 5. By the continuity equation, this means that the CSFBR's jet velocity was half the jet velocity of the Fluoride team, which means that the bottom jet was unable to resuspend flocs that settled at the bottom, causing sludge to build up. The team then replaced the bottom connect of the first reactor with a $\frac{1}{8}$" diameter connect so the jet velocities would be equal for both reactors.

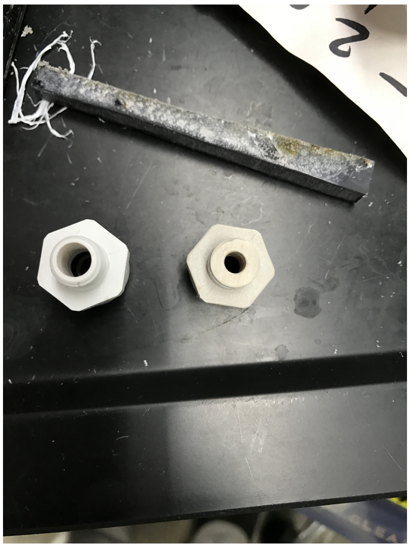

Figure 5: Difference in diameter of failed (left) vs. working (right) bottom connect

The team proceeded to retest the two reactor system at both 5 $\frac{\mathrm{mg}}{\mathrm{L}}$ and 25 $\frac{\mathrm{mg}}{\mathrm{L}}$ of red dye while keeping the concentration of PACl in the reactor at 25 $\frac{\mathrm{mg}}{\mathrm{L}}$ to see if equal jet velocities would decrease the sludge buildup. In addition to testing different ratios, the team also tested the difference between adding PACl immediately into the reactor and delaying the addition. In a test where the PACl was added immediately, at the start of the test, PACl would be continually added to the second reactor while the red dye is added to the first reactor and over time both reactors will form floc blankets with red dye and PACl. During a delayed addition of the PACl, the PACl would be added to the second reactor when the red dye reached the flocculator so that no excess PACl would already be in the second reactor. This was accomplished by starting the PACl pump when the red dye entered the flocculator.

0.1.2 Results and Analysis

When the system from last semester was tested, the team observed settling and sludge buildup at the bottom of the reactor (see Figure 6). As a result, a new bottom geometry was constructed that mimicked the design of the sedimentation tank in an AguaClara plant (see Figure 10 and Figure 11). In the old reactor bottom, the flocs appeared to be settling in a ring around the inflow jet. This could be attributed to the convergence of flocs as they slide down the cone and stick to one another. To address this problem, the new design would have to eliminate the convergence of flocs as they settle out of the floc blanket and are redirected to the jet for resuspension. The new design would theoretically meet this criteria by having an angled plane sloped sharply towards the jet at one side of the reactor base. With a flat surface for the flocs to settle onto and slide down, the flocs would remain at a fixed distance from each other until they reach the jet and are resuspended.

Figure 6: Sludge forming at bottom of reactor 2

Before changing the bottom geometry of the reactors, the team examined two other parameters to explain the settling of flocs: the red dye to PACl ratio and the immediate versus delayed addition of PACl into the system. When the two reactor system was tested with a high concentration of PACl and a low concentration of red dye, specifically 5 $\frac{\text{mg}}{\text{L}}$ of red dye and 25 $\frac{\text{mg}}{\text{L}}$ of PACl, with immediate addition of PACl, the team observed little to no movement in reactor 2 after less than an hour. Settling was observed at the base of reactor 2, with PACl concentrated at the bottom and flocs building up on top of that, as a small jet of influent was flowing up one side of reactor 2 (see Figure 7). Over time, flocs floating within reactor 2 remained stagnant as the high concentration of sticky PACl prevented further circulation. When running the 1:1 ratio test, with 25 $\frac{\text{mg}}{\text{L}}$ of both red dye and PACl and immediate PACl addition, similar buildup of PACl sludge occurred at the base of reactor 2, although there was no blockage farther up the reactor (see Figure 8). This points initially to the suggestion that the primary issue is not the red dye to PACl ratio, but is the resuspension of particles that settle onto the bottom of the reactor, and therefore the new bottom geometry will be necessary. Another possibility was that the PACl is so effective at sticking to itself when it is added immediately, that allowing red dye to pass through the first reactor before letting PACl into the stream (the "delayed" addition method) would allow the PACl to form flocs instead of forming PACl sludge in the second reactor. When this hypothesis was tested, flocs were formed instead of PACl sludge, but the reactor still failed to resuspend particles and a ring of floc sludge formed in both the 1:5 and 1:1 ratio tests (see Figure 9). This further supported the conclusion that the new bottom geometry would be necessary. These results are summarized in Table 2.

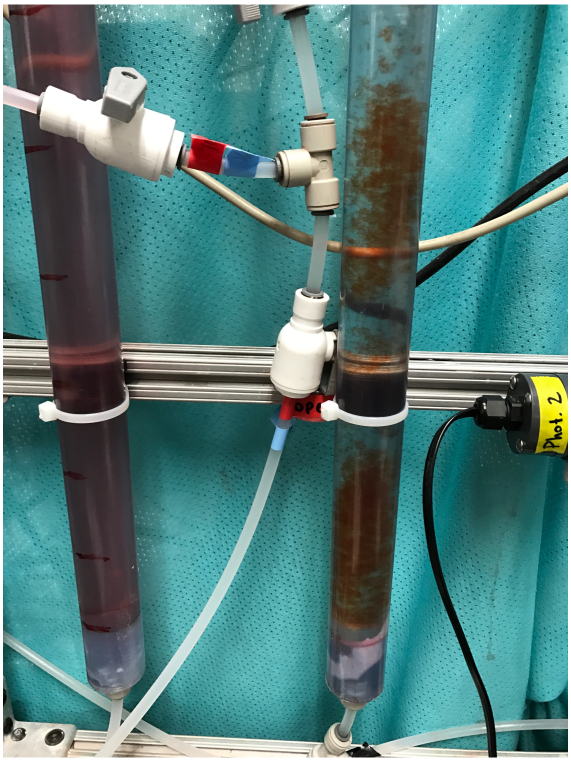

Figure 7: 5 mg/L sludge

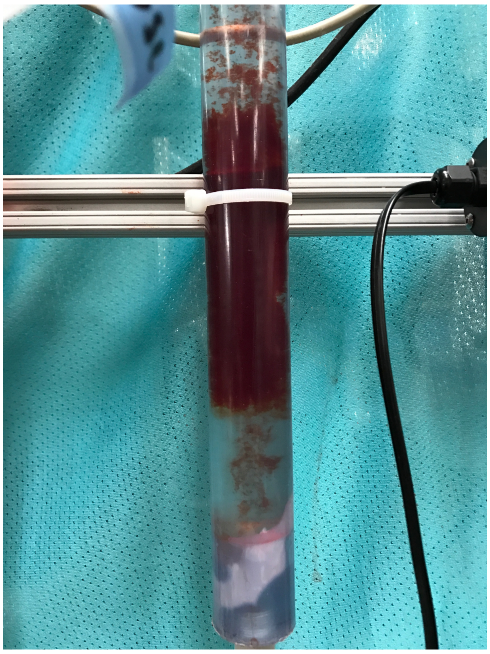

Figure 8: 25 mg/L sludge when the PACl was added immediately

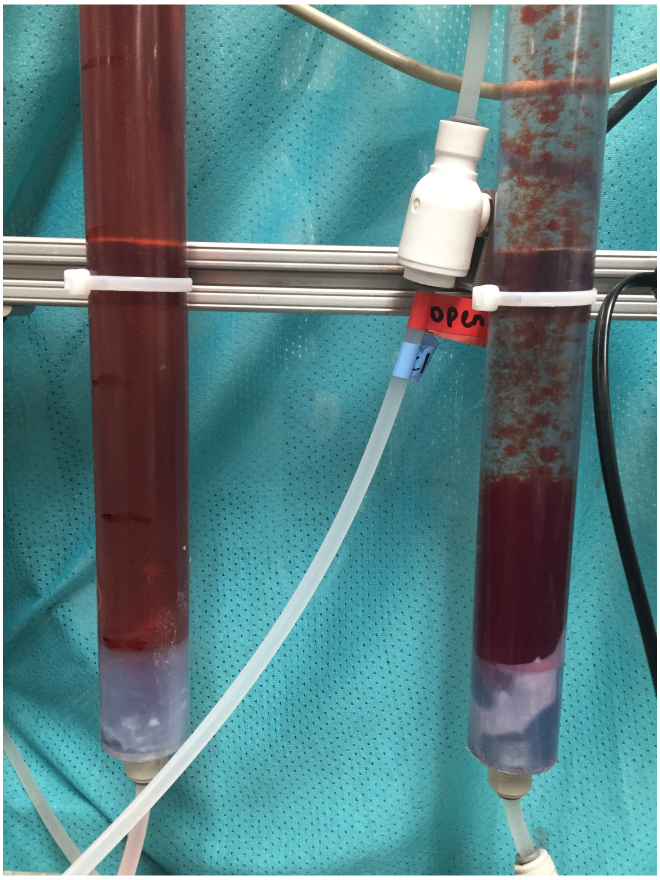

Figure 9: 25 mg/L sludge with delayed addition of PACl

Table 2: Effects of dye concentration and PACl addition on sludge buildup

Concentration of Red Dye in Reactor	PACl addition	Observations
5 mg/L	Immediately	Sludge pockets formed at bottom of reactor and PACl sludge built up at bottom and no resuspension
5 mg/L	Delayed	Sludge pockets formed throughout the reactor and no resuspension
25 mg/L	Immediately	PACl sludge at bottom with red dye sludge above it and no resuspension
25 mg/L	Delayed	Red dye sludge formed at bottom or reactor and no resuspension

0.2 Sloped Bottom Geometry

0.2.1 Procedure

Since the two previous reactors created sludge at the bottom due to the cone shape geometry, the team tested a bottom geometry design that was similar to the jets in the sedimentation tank design in the current AguaClara plants (AguaClara, 2017b). This design featured an angled sloped bottom where the flocs would slide down and then get resuspended at the bottom from the incoming jet of new flocs and water. This would help to prevent settling and sludge buildup at the bottom of the reactor. The geometry is shown in Figure 10 and Figure 11. The team fabricated this design using 1" clear PVC pipe that was 24" long. The bottom of the reactor was heated and molded by inserting a $\frac{1}{4}$" piece of PVC in the bottom until the bottom of the PVC pipe was $\frac{1}{4}$" in width. Then, the PVC pipe was cut at a 30 degree angle and a $\frac{1}{4}$" piece of PVC was glued to the bottom using PVC glue and welded to ensure it was water tight.

Figure 10: Front view of the sloped bottom

Figure 11: Side view of the sloped bottom

After fabrication was complete, the team ran two tests in a one reactor system with the new geometry to see if a floc blanket would form consistently without sludge buildup: one test with 25 $\frac{\text{mg}}{\text{L}}$ of dye and one with 5 $\frac{\text{mg}}{\text{L}}$ of dye. Both tests used 25 $\frac{\text{mg}}{\text{L}}$ of PACl.

0.2.2 Results and Analysis

Since the cone bottom geometry failed at providing resuspension in the bottom of the reactor, the sloped bottom geometry was proposed, fabricated, and then tested. When the new geometry was tested at an upflow velocity of 1 $\frac{\text{mm}}{\text{s}}$, there was circulation of flocs within the entire reactor and a floc blanket was successfully created within 10-15 minutes (see Figure 12 and Figure 13). These results showed significant improvement compared to previous tests with the cone bottom geometry, particular from Fall 2016 semester, in which the two reactor system had to be run at upflow velocities closer to 1.5 $\frac{\text{mm}}{\text{s}}$ in order to get any reasonable amount of resuspension in the base of the reactor. This suggests that reactors designed with this new geometry will be more successful in creating a fluidized bed than the previous bottom geometry design.

Figure 12: Recirculation with new bottom geometry with 5 mg/L of dye

Figure 13: Recirculation with new bottom geometry with 25 mg/L of dye

0.3 Effect of Dye to PACl Ratio

0.3.1 Procedure

With the new bottom geometry successfully recirculating flocs at the bottom of the reactor, the team proceeded to test the effect of different dye to PACl ratios on the efficiency of a one reactor versus a two reactor system. The variation in dye to PACl ratio was intended to test whether an excess of PACl would provide better or worse removal of red dye than an equal ratio. The team ran three tests: two two reactor tests with a 1:1 and 1:5 ratio of dye to PACl, respectively, and a one reactor test with a 1:1 ratio of dye to PACl. As explained in the results, the 1:5 ratio two reactor test failed due to sludge buildup and thus it was unnecessary to run a one reactor test knowing that it would fail. The concentration of PACl in the reactor stayed constant at 25 mg/L and the dye concentrations used were 25 mg/L and 5 mg/L. In all of these tests, the PACl was added immediately into the second reactor due to ease of operation since the previous tests found that the timing of PACl addition was not the cause of sludge buildup. The difference between using two reactors and one reactor was more accurately tested since no sludge built up at the bottom of the reactor due to the improved bottom geometry. The stock concentrations that were used to provide appropriate concentrations into the reactor using the ProCoDA file and MathCAD files for flow rates were 250 mg/L of red dye and 250 mg/L of PACl for the 1:1 ratio test and 50 mg/L of red dye and 250 mg/L of PACl for the 1:5 ratio test.

0.3.2 Results and Analysis

Using the reactors with the sloped bottom geometry, the effluent concentration of the one reactor test with a 1:1 dye to PACl ratio continued to rise and did not reach an asymptote within 20 hours. This phenomenon was also seen by the High Rate Sedimentation subteam and there is no current conclusion or explanation for why this was happening. However, the effluent concentration remained below 1.8 $\frac{\text{mg}}{\text{L}}$ within 20 hours (see Figure 14). For the same concentrations of dye and PACL in the two reactor system, the effluent concentration again was not constant, but remained below 3 $\frac{\text{mg}}{\text{L}}$ within 24 hours. The concentration of water exiting reactor 1 showed little reduction in concentration from the influent concentration (25 $\frac{\text{mg}}{\text{L}}$) as seen in Table 3, which suggests that this additional reactor does not significantly provide increased removal of red dye from the system (see Figure 15). A summary of the results for the one and two reactor tests for different dye to PACl ratios are shown in Table 3. The hypothesis for this counterintuitive phenomenon is that the PACl particles get saturated with dye in reactor 2 so that when they are added into the reactor 1, no additional dye would adsorb to the PACl before the flocs exit through the weir.

In the two reactor test with a 1:5 dye to PACl ratio, the system failed after about 7 hours (see Figure 16). In reactor 2, blocks of PACl and dye formed, preventing the formation of a good fluidized bed. The hypothesis is that with such an excessive amount of PACl in the system, PACl was adsorbing to itself so strongly that it was forming a thick gel or sludge that was too dense to be recirculated and rather it just accumulated on top of each other causing sludge buildup. This sludge buildup is eventually what caused the failure of the reactor. Since lower dissolved species to coagulant ratios are used in the field, the subteam must find a way to resolve this issue.

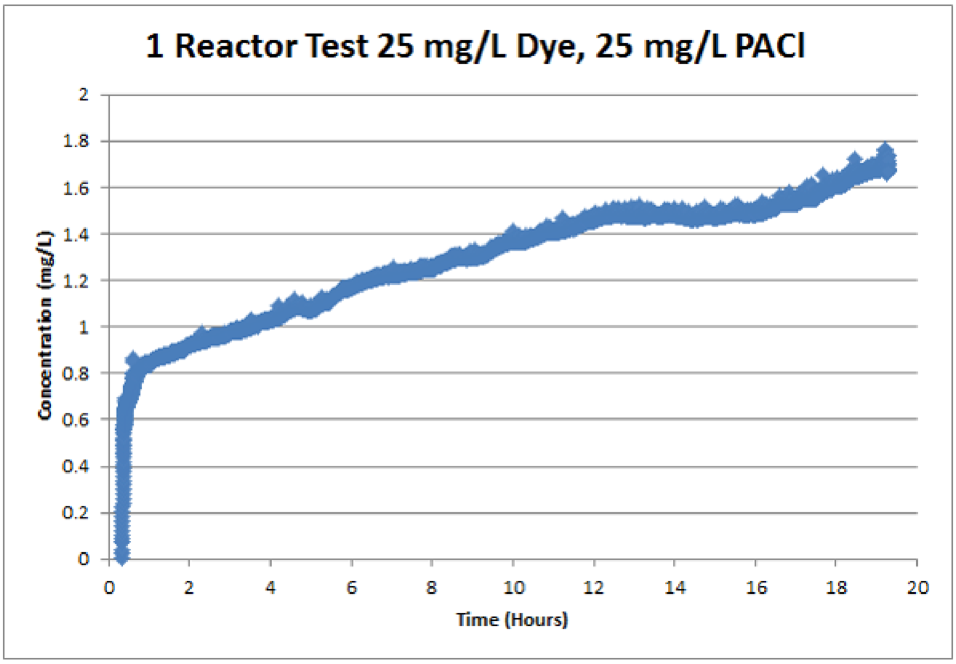

Figure 14: One-reactor test 25 mg/L dye, 25 mg/L PACl

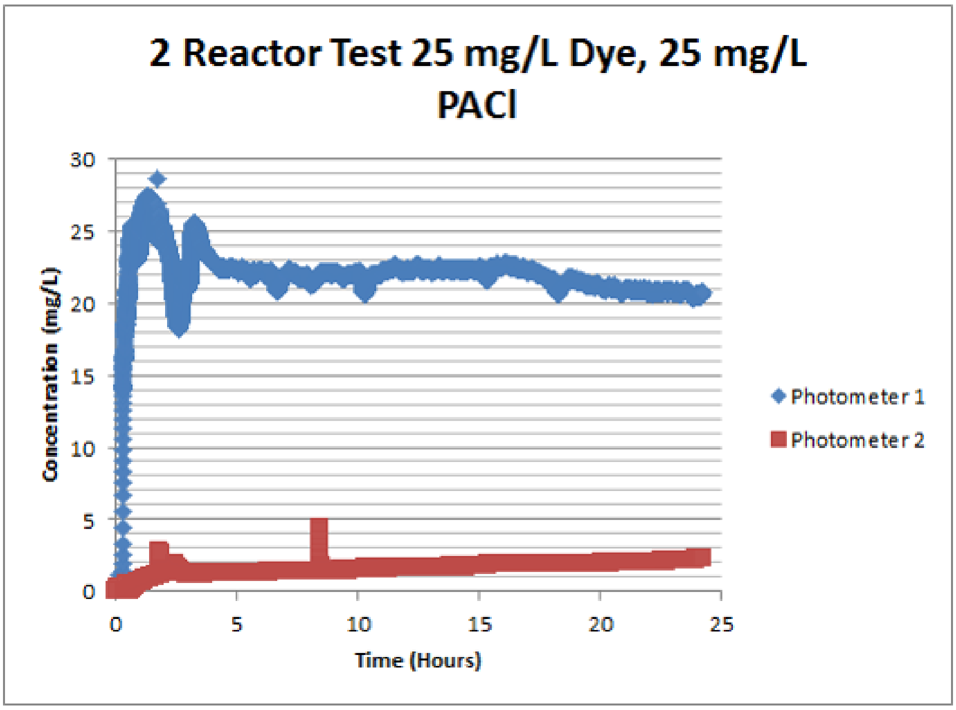

Figure 15: Two-reactor test 25 mg/L dye, 25 mg/L PACl

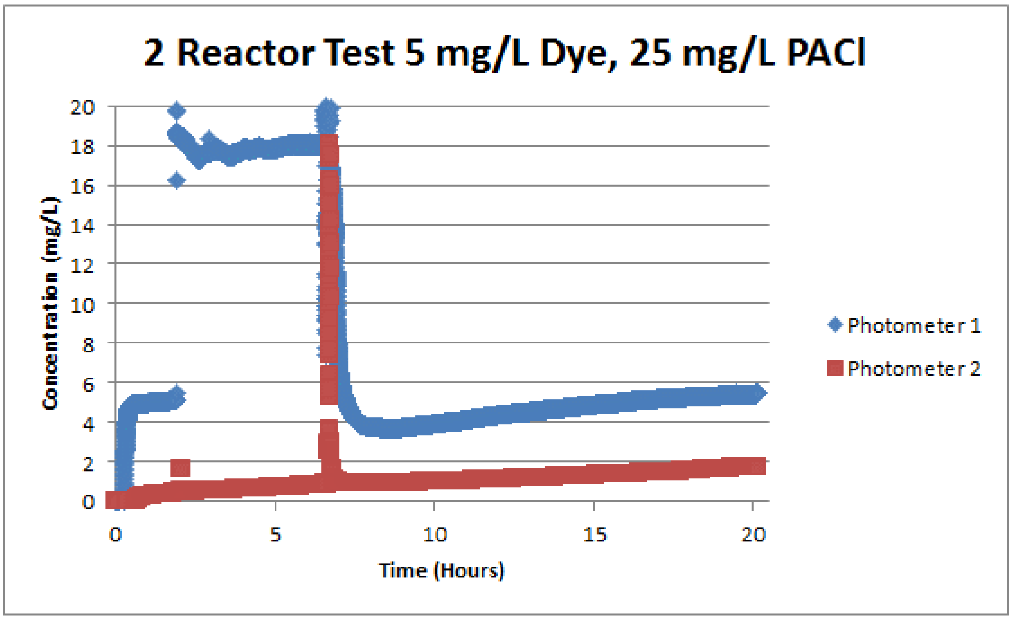

Figure 16: Two-reactor test 5 mg/L dye, 25 mg/L PACl

Table 3: Effects of dye to PACl ratio on red dye removal percentage

Reactor Test	Reactor Conc Red Dye	Reactor Conc PACl	Reactor 2 Effluent Red Dye Conc	Reactor 1 Effluent Red Dye Conc	Run Time
1 Reactor	25 mg/L	25 mg/L	1.7 mg/L	N/A	20 hours
2 Reactor	25 mg/L	25 mg/L	2 mg/L	23 mg/L	25 hours
2 Reactor	5 mg/L	25 mg/L	1.9 mg/L	5 mg/L	20 hours

There is no evidence through these experiments that suggests that one reactor is better than two for red dye removal as seen in Table 3. The one reactor and two reactor test for a dye to PACl ratio of 1:1 shows no significant difference in the effluent concentration and there is no significant removal happening in the first reactor. In the context of removal of dye at the larger scale, this data suggests that using a low concentration of dye compared to PACl could cause sludge problems and using equal concentrations has no need for a second reactor. Therefore, a second reactor is not recommended for dye removal in the field. However, more testing can be done to check the minimum dosage of PACl necessary to provide sufficient dye removal and cause no sludge problems.

0.4 Testing Fluoride Removal with New Reactors

0.4.1 Procedure

In order to reach a more definitive conclusion as to which system was more effective in removing fluoride, the red dye used in previous experiments was replaced with fluoride. The photometers were replaced with one fluoride probe that was used to measure the effluent of reactor 2. The fluoride probe was re-calibrated before every test and was used to compare the percent removal between a one reactor system and two reactor system. As suggested by results of preliminary experiments from the Spring 2016 semester, clay is necessary for fluoride removal because fluoride and PACl are unable to create dense flocs that settle out (Longo et al., 2016). Therefore, clay was added into the influent water stream from an additional stock tank for these tests. The team ran four tests with two different variations of PACl and clay concentrations using both the one reactor and two reactor system while keeping a constant concentration of 10 $\frac{mg}{L}$ fluoride in the reactor. At first, the team replicated a test done in Spring 2016 (Longo et al., 2016) using 1200 $\frac{mg}{L}$ of clay and 50 $\frac{mg}{L}$ of PACl, but later reduced the concentrations of PACl and clay to 25 $\frac{mg}{L}$ and 100 $\frac{mg}{L}$, respectively. High concentrations of PACl were used because the hypothesis is that dissolved ions such as fluoride require a higher dosage of PACl than particles that can settle out such as clay.

The PACl and clay concentrations for the second test were chosen based on the calculation of a gamma value. Gamma value represents the percentage of clay particle surface area that is covered by coagulant. It is a function of clay concentration, coagulant concentration, type of coagulant, diameter of flocculator tube, diameter of clay particles, and the geometry of clay particles (see Figures 17 and 18) AguaClara (2017a). The initial aim of the team was to conduct experiments with a gamma value approximately equal to two and one to test the relationship between gamma and fluoride removal percentage.

$$\Gamma_{Coag}(C_{Clay}, C_{Al}, EN_{Chem}, D_{Tube}, D_{Clay}, \Pi_{HD}) := 1 - \exp\left(\frac{-\phi_{Floc.0}\left(C_{Al}, 0\frac{mg}{L}, EN_{Chem}\right)\cdot D_{Clay}}{\phi_{Floc.0}\left(0\frac{mg}{L}, C_{Clay}, EN_{Chem}\right)\cdot D_{nc}(EN_{Chem})}\cdot\frac{1}{\pi}\cdot\frac{\Pi_{AClayATotal}(C_{Clay}, D_{Clay}, D_{Tube}, \Pi_{HD})}{\Pi_{ClaySphere}(\Pi_{HD})}\right)$$

Figure 17: Gamma equation as coded in MathCAD

$$\phi_{Floc.0}(C_{Al}, C_{Clay}, EN_{Chem}) := \left| \begin{array}{l} \phi_{Floc.0} \leftarrow \frac{C_{Precipitate}(C_{Al}, EN_{Chem})}{\rho_{AlOH3}} + \frac{C_{Clay}}{\rho_{Clay}} \text{ if } EN_{Chem} = 0 \\ \phi_{Floc.0} \leftarrow \frac{C_{Precipitate}(C_{Al}, EN_{Chem})}{\rho_{PACl}} + \frac{C_{Clay}}{\rho_{Clay}} \text{ otherwise} \\ \text{return } \phi_{Floc.0} \end{array} \right.$$

$$\Pi_{ClaySphere}(\Pi_{HD}) := \left(\frac{1}{2} + \Pi_{HD}\right)\cdot\left(\frac{2}{3\cdot\Pi_{HD}}\right)^{\frac{2}{3}}$$

$$\Pi_{AClayATotal}(C_{Clay}, D_{Clay}, D_{Tube}, \Pi_{HD}) := \frac{1}{1 + \frac{2\cdot D_{Clay}}{3\cdot D_{Tube}\cdot\Pi_{ClaySphere}(\Pi_{HD})\cdot\frac{C_{Clay}}{\rho_{Clay}}}}$$

Figure 18: Secondary equations necessary to calculate gamma as coded in MathCAD

The system setup was slightly different than the setup used for red dye. The red dye stock tank was replaced with a clay stock tank that ran through the same pump head as the red dye. A fluoride stock tank was also added to the system and ran through the water pump head (see Figure 19). The flow rate of water was halved because of the addition of the fluoride line so that the same raw water flow rate was maintained from the dye setup to the fluoride setup to maintain the appropriate upflow velocity. The fluoride in the stock tank had a concentration of 25 $\frac{mg}{L}$ and the influent fluoride concentration was measured with the fluoride probe after calibration. The stock tank concentrations of PACl and clay were 10 times their respective reactor concentrations.

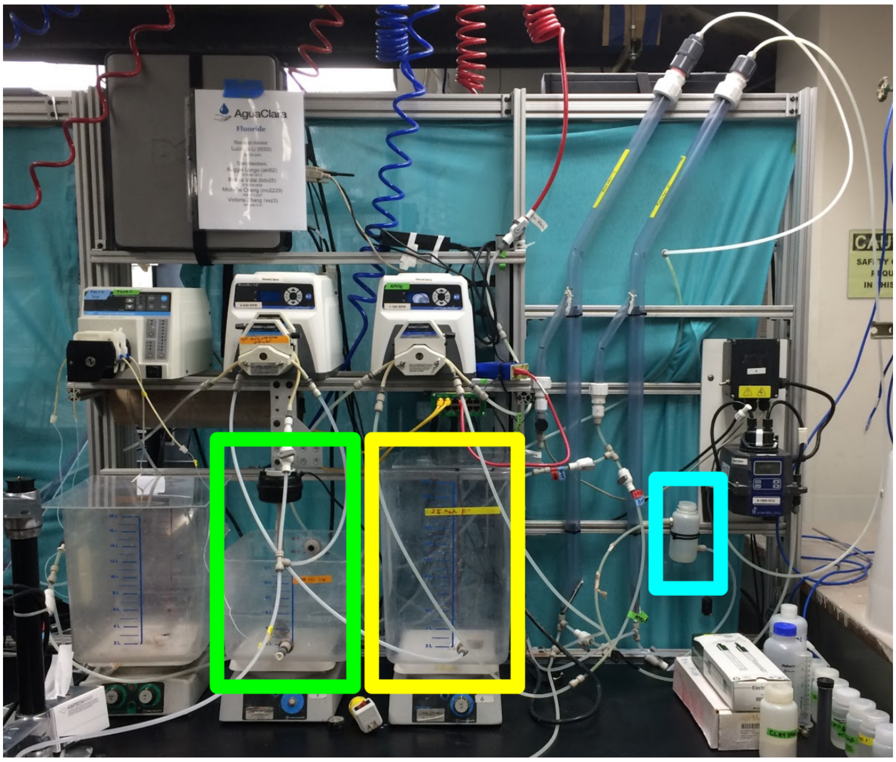

Figure 19: Changes made to the system setup for fluoride testing including clay stock tank (green), fluoride stock tank (yellow) and fluoride probe (blue)

0.4.2 Results and Analysis

The one reactor and the two reactor tests for the 1200 $\frac{\text{mg}}{\text{L}}$ clay and 50 $\frac{\text{mg}}{\text{L}}$ PACl in the reactor provided sufficient removal of fluoride, which fell well below the WHO standard for fluoride in drinking water (1.5 $\frac{\text{mg}}{\text{L}}$). The two reactor system removed approximately 97.3% of the fluoride, with an effluent concentration of only 0.24 $\frac{\text{mg}}{\text{L}}$ (Figure 20). The one reactor test had similar results, removing approximately 95.0% of the fluoride, producing an effluent concentration of 0.42 $\frac{\text{mg}}{\text{L}}$ (Figure 21). The two reactor test for concentrations of 25 $\frac{\text{mg}}{\text{L}}$ of PACl and 100 $\frac{\text{mg}}{\text{L}}$ of clay provided decent removal of fluoride, just barely meeting WHO standards with 1.22 $\frac{\text{mg}}{\text{L}}$ effluent fluoride and 81.3% removal (Figure 22). However, the one reactor system failed to fall within WHO drinking water standards (although it met EPA standards), providing 77.5% removal and 2.00 $\frac{\text{mg}}{\text{L}}$ effluent fluoride concentration (Figure 23). These results are summarized in Figure 24.

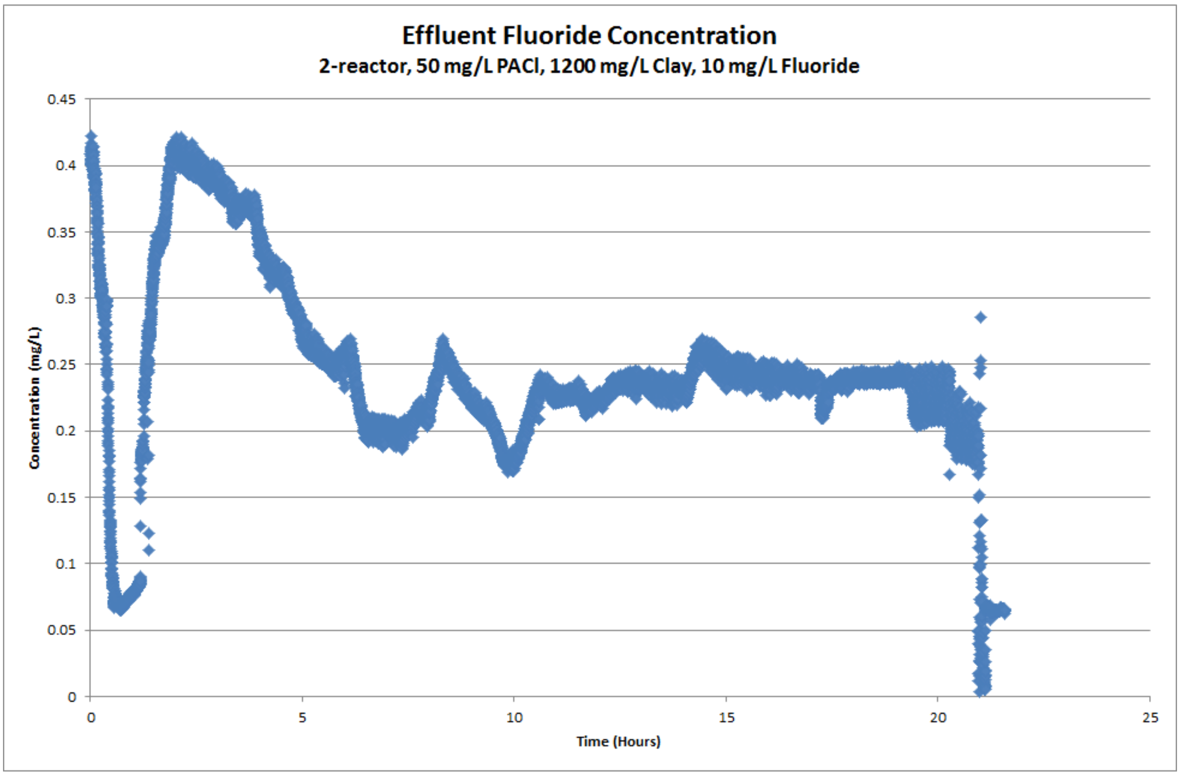

Figure 20: Two-reactor test with 50 mg/L PACl, 1200 mg/L clay

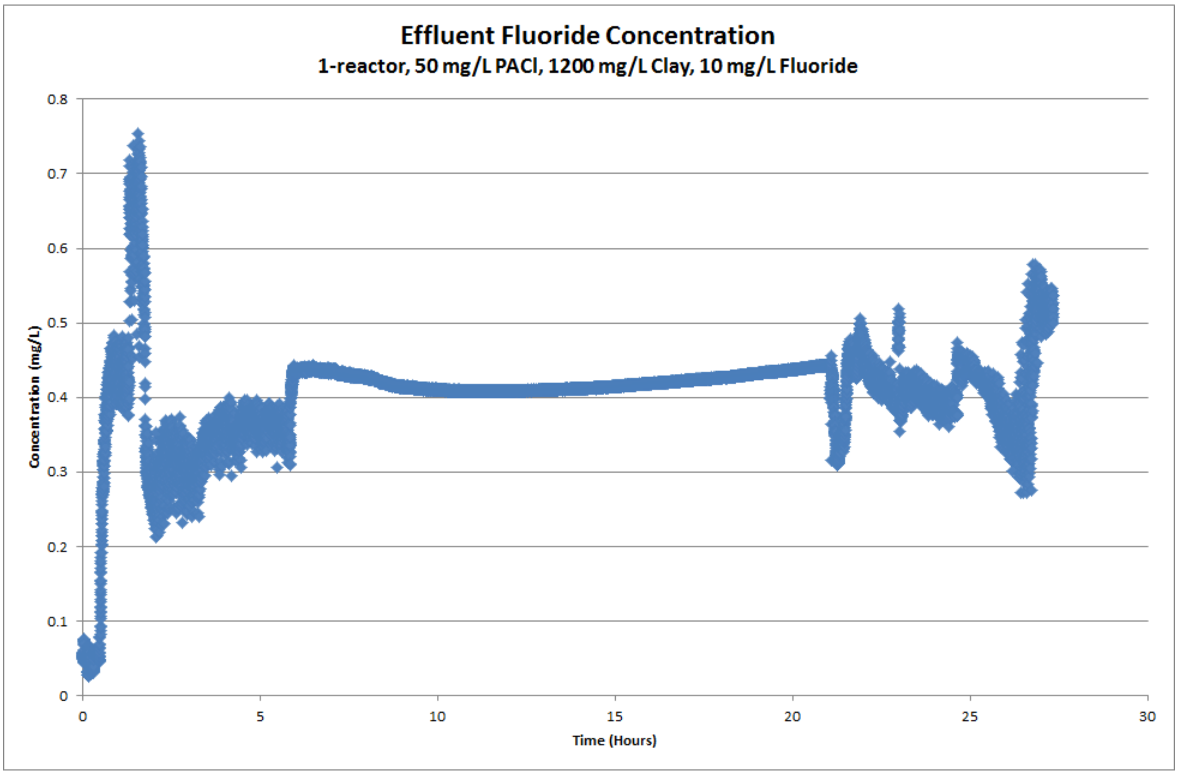

Figure 21: One-reactor test with 50 mg/L PACl, 1200 mg/L clay

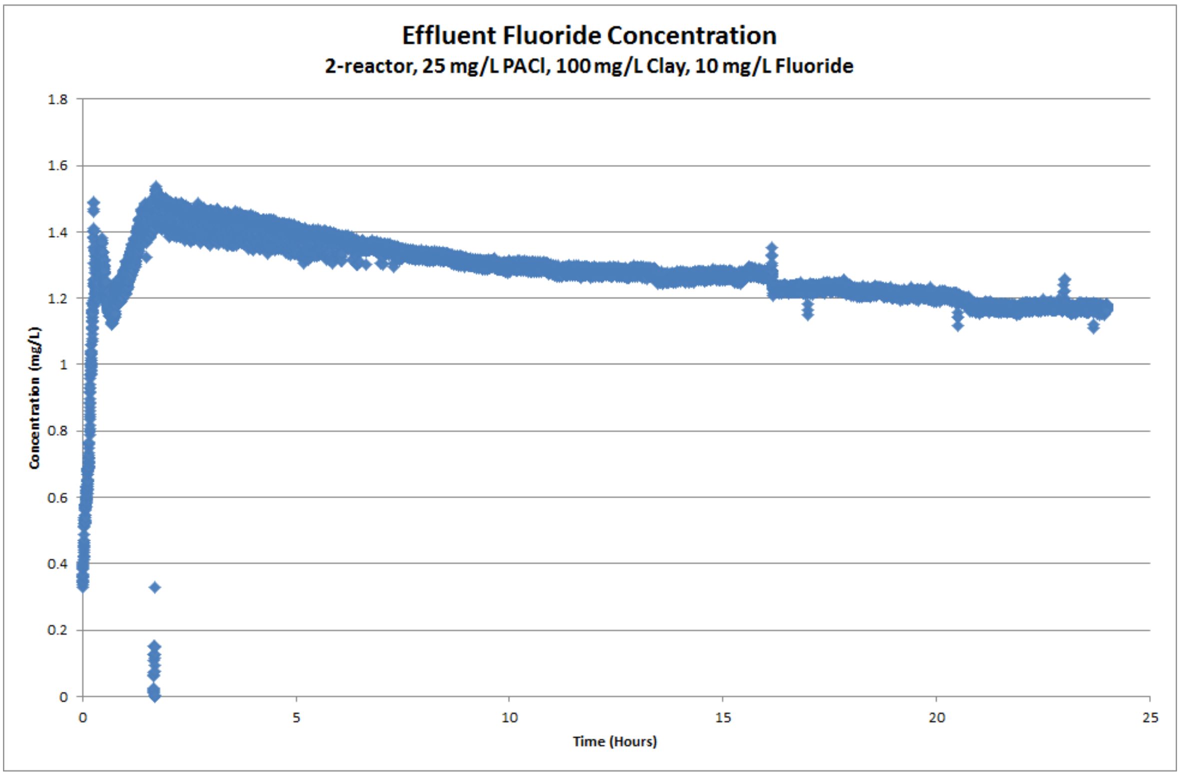

Figure 22: Two-reactor test with 25 mg/L PACl, 100 mg/L clay

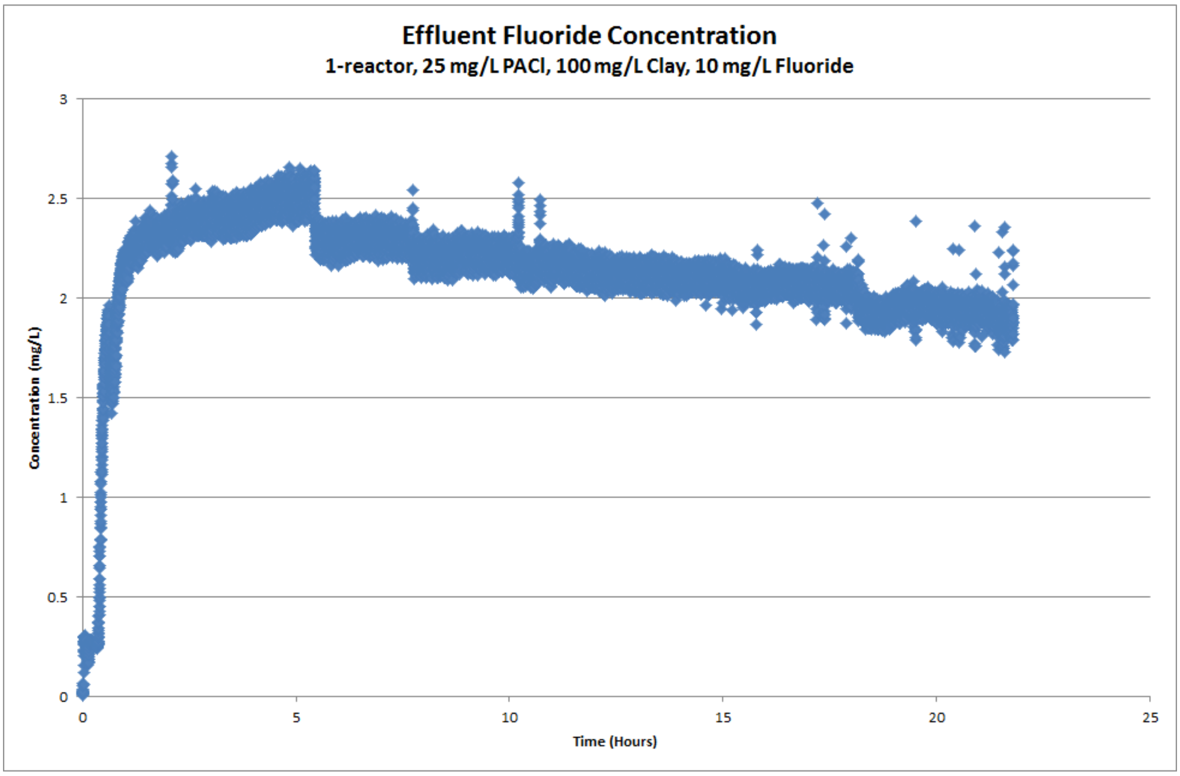

Figure 23: One-reactor test with 25 mg/L PACl, 100 mg/L clay

Test Type	Influent Fluoride	Effluent Fluoride	Influent PACl Concentration	Influent Clay Concentration	Percent Removal
2-reactor	8.81	0.24	50	1200	0.972755137
2-reactor	6.534	1.22	25	100	0.813284359
1-reactor	8.358	0.42	50	1200	0.949748744
1-reactor	8.90	2.00	25	100	0.775331386

Figure 24: Percent Removal with different PACl to clay ratios

At this point, more experiments at lower dosages of clay and PACl and more replications of previous experiments need to be completed to provide a better understanding as to whether a two reactor system can perform significantly better than a one reactor system. In addition, more research needs to be done to test the importance of clay and PACl in the removal of fluoride. PACl costs more than clay so if PACl is the more important factor, then the operating costs are dependent on the dosing of PACl and experimentation will be done to find the optimal and minimum dosage. However, if clay is the more important factor, then addition of clay into a full-scale AguaClara system may cause additional problems to solve due to the natural settling of clay.

Conclusions

At the start of the semester, the subteam was unsure if last semester's system would be able to effectively remove dissolved species like originally hoped. Although the reactor had been very effective when red dye and PACl were mixed in equal concentrations, in reality, the concentration of fluoride would be much lower than that of coagulant, perhaps by a tenfold factor. However, when the team performed tests with higher concentrations of PACl, PACl sludge accumulation prevented the collection of data. Thus, a new series of reactors with sloped bottom geometry was created in order to facilitate better floc resuspension in the presence of "sticky", unsaturated PACl. This system provided the necessary recirculation of PACl and clay in order to effectively remove fluoride below WHO regulatory levels. After running both single-reactor and two-reactor tests, the subteam found a slightly higher percent removal in a two-reactor system. However, more tests will be required to determine if this difference is statistically significant. With less than 5% difference, it seems unlikely that this will turn out to be significant. In this case, utilizing additional resources to build two reactor systems in future AguaClara plants will probably not be the most efficient way to maximize dissolved particle removal, however future testing can provide the necessary conclusions for this hypothesis. Recommended experiments include a high PACl and low clay dosage as well as a high clay and low PACl dosage to test which compound has more impact on fluoride removal.

Future Work

In future semesters, the variables PACl concentration and clay concentration should be examined in further detail with regard to fluoride removal, focusing on reducing cost of operation while maintaining the WHO standard of removal that was obtained this semester. These tests should include examining the effect of varied gamma values. Testing of one reactor versus two reactor systems should continue so that the difference in removal efficiency can be more accurately quantified with a larger volume of data. Running multiple iterations with the same variables will allow average results to be established. After quantifying fluoride removal efficiency, the effects of pH on fluoride removal should be examined. The team should also begin designing a gravity-powered, scaled-up system for implementation in the field.

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Semester Schedule

Task Map

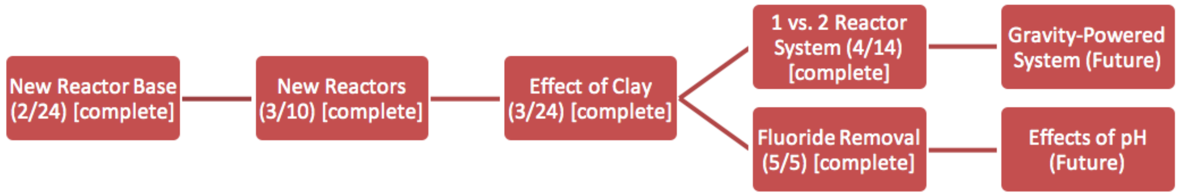

Figure 25: Fluoride subteam task map Spring 2017

Task List

1. ✓Design and fabricate a new reactor base (2/24/17) - Briana. Floc blanket settling at the base of reactors has been a problem with higher concentration floc blankets. Creating a new base geometry may resolve this problem.

2. ✓Fabricate and test reactors with sloped bottom (3/10/17) - Victoria. Fabricate two complete reactors with the new sloped bottom geometry and test whether they can consistently form a good floc blanket with various concentrations of red dye and PACl.

3. ✓Determine the effect of adding clay to PACl solution (3/24/17) - Michelle. Run tests in with clay added to PACl stock solution to determine if it allows floc blankets to form better by preventing PACl particles from adsorbing to other PACl particles.

4. ✓Test red dye removal using one- and two-reactor systems (4/14/17) - Auggie. Run experiments with identical red dye and PACl concentrations and upflow velocity in the 1 and 2 reactor system to determine whether a 2 reactor system can produce as good or better removal efficiency while using less PACl.

5. ✓Test fluoride removal (5/5/17) - Briana. Using the most efficient system as determined by the preceding tests, quantify the removal efficiency of fluoride in that system.

6. Determine effects of pH on fluoride removal (Next Semester) - Team Member. Vary the pH of the influent water stream to determine the effects on fluoride removal.

7. Design a gravity-powered system (Next Semester) - Team Member. Designing a gravity-powered system will allow this method to be used in the field where electricity is not readily available.

Report Proofreader: Briana Li-Vidal

Manual

Experimental Methods

Red Dye Experimentation

1. Open and close all the appropriate valves as labeled with red and blue tape. The red tape is to run a two reactor test and the blue tape is to run a one reactor test.
2. Turn on Just Water process in ProCoDA and fill system completely with water. *Make sure to open the waste line*
3. Continue to run water until both photometers give readings less than 0.20 mg/L and turbidimeter is less than 0.4 NTU.
4. Fill stock tanks with appropriate concentration of PACl and dye, and make sure to have enough stock to run for 24 hours.
5. Write a text file in ProCoDA saying ”Start Test” and then change the process to the ON state.
6. Put the waste pump at the appropriate RPM as calculated from ProCoDA
7. Recheck everything is running how it should be.
8. At the end of the test change the process to the OFF state.
9. Clean the reactor using the ”Cleaning Procedure” after the experiment is completed.

Cleaning Procedure

(a) Put a piece of sponge in the tube between the flocculator and PACl insert.
(b) Run a high velocity jet through the tube to purge the flocculator of excess dye.
(c) Drain both reactors through the valves at the bottom of the reactors.
(d) Flush water through both reactors until no dye remains in the system.
(e) If there is not a noticeable amount of buildup, (a) and (b) can be skipped.

Fluoride Experimentation

1. Open and close all the appropriate valves as labeled with red and blue tape. The red tape is to run a two reactor test and the blue tape is to run a one reactor test.
2. Turn on Just Water process in ProCoDA and fill system completely with water. *Make sure to open the waste line and the fluoride line and close the fluoride valve as to not pump fluoride into the system during backwash*
3. Continue to run water until turbidimeter is less than 0.6 NTU and fluoride concentration less than 0.5 mg/L. Make sure fluoride probe bottle does not overflow with water.
4. Fill stock tanks with appropriate concentration of PACl, fluoride, and clay, and make sure to have enough stock to run for 24 hours.
5. Empty the bucket at the bottom and make sure it doesn't overflow through the length of the test.
6. Write a text file in ProCoDA saying ”Start Test” and then change the process to the ON state.
7. Run the waste pump at the appropriate RPM as calculated from ProCoDA.
8. Recheck everything periodically to ensure it is running how it should be and there are no water leaks.
9. At the end of the test change the process to the OFF state.
10. Clean the reactor using the ”Cleaning Procedure” after the experiment is completed.

Cleaning Procedure

(a) Put a piece of sponge in the tube between the flocculator and PACl insert.
(b) Run a high velocity jet through the tube to purge the flocculator of excess clay buildup.
(c) Drain both reactors through the valves at the bottom of the reactors.
(d) Flush water through both reactors until no clay remains in the system.
(e) If there is not a noticeable amount of buildup, (a) and (b) can be skipped.

Experimental Checklist

1. Waste line is open (System will explode if this is not open)
2. No leaks anywhere in system
3. Pumps running at the correct RPM (Check ProCoDA)

ProCoDA Method File

ProCoDA is a process control system that was developed by Monroe Weber-Shirk in order to set process parameters through a computerized system. It can be adjusted to different system states that control the system pumps depending on what flow rates are desired. Additionally, ProCoDA collects the data from probes, allowing for compilation of dye concentration data.

To begin the ProCoDA method file, three states were made: ON and OFF and Just Water. In the OFF state, all the valves were closed and no pumps were on. In the ON state, all the pumps were ON and all valves were opened, in the Just Water state, only the water valve was open. ProCoDa turned this pump on and off via a normal valve control, so long as the pump was already set to a proper flow rate. The system was set to run on Manual setting, as a proper run time had not yet been determined.

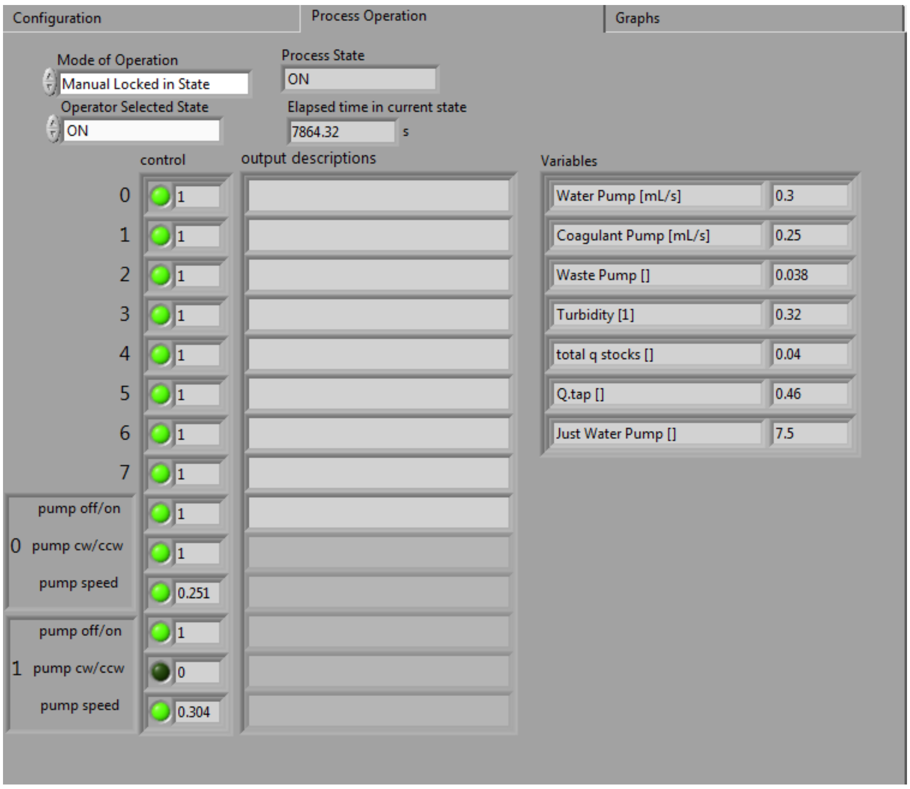

Figure 26: ScreenShot of ProCoDA Panel

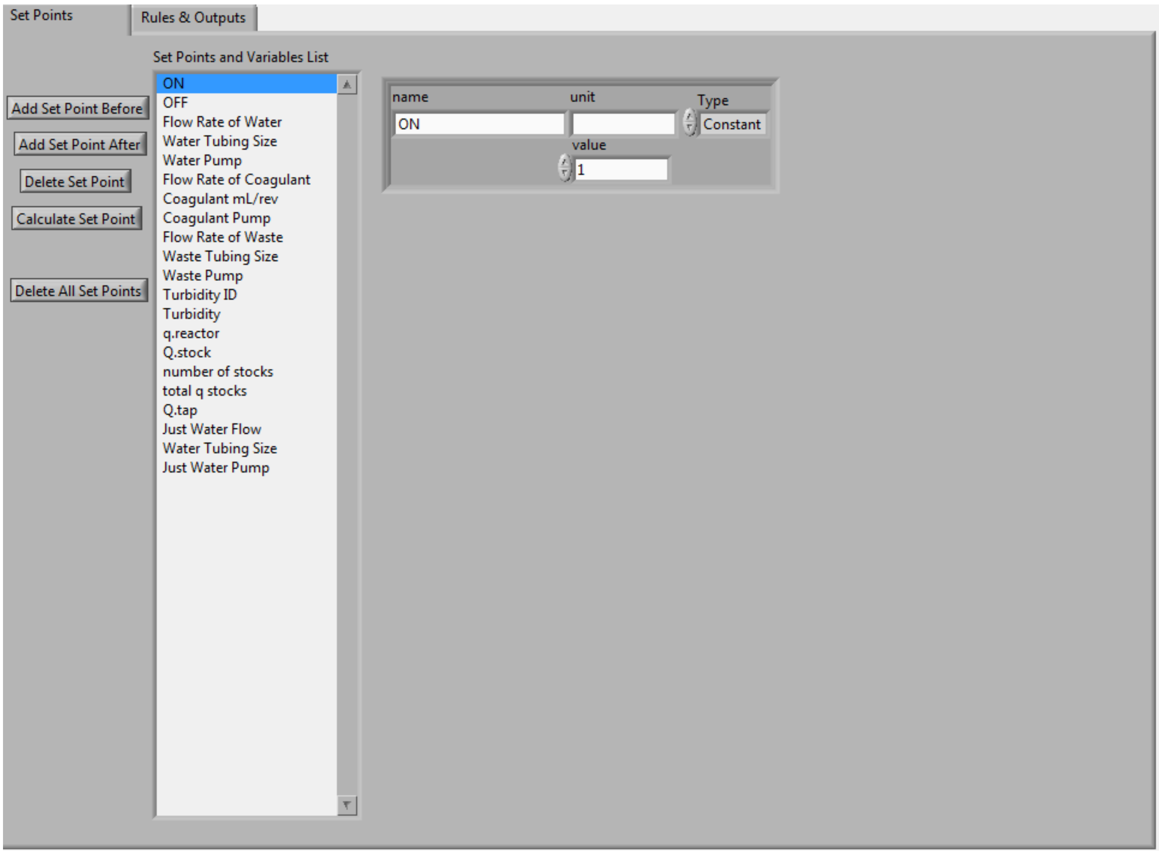

Figure 27: ScreenShot of ProCoDA Set Points

The method file was set to control the revolutions per minute (RPM) of the PACl/dye pump and the tap water pumps. This was done using the peristaltic pump ProCoDA file available in the AguaClara server, and inputs for desired flow rate and tubing size. For the PACl and dye pump heads, inputs of $\frac{\text{mL}}{\text{rev}}$ and flow rate were needed to calculate RPM since microtubing was used, and for the water pump head, tubing ID and flow rate were needed to calculate RPM. The set points used for the method file included a water pump set point for the water pump RPM and a floc pump set point for the PACl/dye pump RPM.

Table 4: List of variables for ProCoDA

Set Point	Definition	Value
Water Flow Rate	Flow rate of water through the system	Variable
Water Tubing ID	Pump tubing size	16
Water Pump	Water pump RPM	Variable
Floc Flow Rate	Flow rate of PACl and dye through the system	Variable
Floc mL/Rev	Volume of water per revolution of the pump	0.012195
Floc Pump	Dye and PACl pump RPM	Variable