Advancing the Implementation and Adoption of Urine Diversion Systems in

Commercial and Institutional Buildings in the United States: A Focus on

Control of Urea Hydrolysis

by

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ABSTRACT

This dissertation focused on the implementation of urine diversion systems in commercial and institutional buildings in the United States with a focus on control of the urea hydrolysis reaction. Urine diversion is the process by which urine is separately collected at the source in order to realize system benefits, including water conservation, nutrient recovery, and pharmaceutical removal. Urine diversion systems depend greatly on the functionality of nonwater urinals and urine diverting toilets, which are needed to collect undiluted urine. However, the urea hydrolysis reaction creates conditions that lead to precipitation in the fixtures due to the increase in pH from 6 to 9 as ammonia and bicarbonate are produced.

Chapter 2 and Chapter 3 describes the creation and use of a cyber-physical system (CPS) to monitor and control urea hydrolysis in the urinal testbed. Two control logics were used to control urea hydrolysis in realistic restroom conditions. In the experiments, acid was added to inhibit urea hydrolysis during periods of high and low building occupancy. These results were able to show that acid should be added based on the restroom use in order to efficiently inhibit urea hydrolysis.

Chapter 4 advanced the results from Chapter 3 by testing the acid addition control logics in a real restroom with the urinal-on-wheels. The results showed that adding acid during periods of high building occupancy equated to the least amount of acid added and allowed for urea hydrolysis inhibition. This study also analyzed the bacterial communities of the collected urine and found that acid addition changed the structure of the bacterial communities.

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Chapter 5 showed an example of the capabilities of a CPS when implemented in CI buildings. The study used data mining methods to predict chlorine residuals in premise plumbing in a CI green building. The results showed that advance modeling methods were able to model the system better than traditional methods. These results show that CPS technology can be used to illuminate systems and can provide information needed to understand conditions within CI buildings. To Gala Beatriz Del Castillo and Gladys Ferro

Because representation matters

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CHAPTER 1

INTRODUCTION

In recent years, water scarcity and drought has affected large areas of the southwest and southern United States and it is expected that climate change will exacerbate water issues across the country (Georgakakos et al., 2014). As cities face variable water quantity, policymakers have implemented programs for water conservation and have explored new water sources that can be added to their portfolios, such as indirect potable reuse (National Research Council, 2012). As one such solution, drinking water treatment plants have invested in source water protection programs to ensure that available water supplies are treatable to drinking water standards (Price & Heberling, 2018). The building construction industry has also responded to calls for water conservation and resource efficiency by creating certification programs focused on green design, such as the U.S. Green Building Council's Leadership in Energy and Environmental Design (LEED) program (US Green Building Council, 2019). Approximately 17% of public water supplies in the United States are used in commercial and industrial (CI) buildings (US Environmental Protection Agency, 2012). This includes office buildings, hotels, restaurants, hospitals, and schools. Within the wide variety of CI buildings, the largest amount of water (approximately 30%) is used for restrooms and domestic use (US Environmental Protection Agency, 2012). Therefore, water savings within restrooms are an area of focus for those seeking to lower their environmental impact.

1

Urine diversion has been proposed as a process for source water protection, via diversion of nutrients and micropollutants, and water conservation, via the collection of urine without the use of flush water in urinals and toilets (Larsen & Gujer, 1996). Urine diversion is the process by which urine is separately collected at the source in order to capture its many benefits, with one being water conservation. Landry and Boyer found that campus wide implementation of urine diversion at the University of Florida would equate to potable water savings of 69,000,000 gallons and \$231,000 per year for the university, while reducing energy and resource demands at the water treatment facility (Landry & Boyer, 2016). Additionally, removing the urine stream from wastewater collection dramatically changes the composition of wastewater entering wastewater treatment plant, which provides the opportunity to reduce energy and resource consumption (Jimenez, Bott, Love, & Bratby, 2015). This is largely due to the fact that urine contributes 80% of the nitrogen (N) and 50% of the phosphorus (P) to wastewater treatment plants, while only contributing to 1% of the volumetric flow (Wilsenach & van Loosdrecht, 2006). Rauch et al. also showed that diverting human urine from wastewater collection could reduce peak ammonia loads by as much as 30%, eliminating the need for expansion of centralized wastewater treatment infrastructure (Rauch, Brockmann, Peters, Larsen, & Gujer, 2003). Hence, urine diversion becomes a solution for the inefficient removal of nutrients during wastewater treatment that leads to eutrophication of natural water ways (Anderson, Glibert, & Burkholder, 2002; Lapointe, Herren, Debortoli, & Vogel, 2015; Van Drecht, Bouwman, Harrison, & Knoop, 2009). Once urine is collected, nutrient recovery technologies can be used to recover a sustainable source for fertilizers and other N, P, and K products. One of the most studied technologies for nutrient

recovery from human urine is struvite precipitation for phosphorus recovery, which is a relatively simple technology requiring the addition of magnesium at a 1:1 molar ratio to precipitate NH₄MgPO₄·6H₂O (Etter, Tilley, Khadka, & Udert, 2011; Kabdaşlı et al., 2006; Ronteltap, Maurer, & Gujer, 2007; K. M. Udert, Larsen, & Gujer, 2003b). Moreover, due to the aforementioned benefits of urine diversion, technology development has led to the creation of systems for recovery of nutrients via a wide variety of physical, chemical, and electrochemical systems (Patel, Mungray, & Mungray, 2020).

As stated above, urine diversion also provides the opportunity to remove pharmaceuticals and micropollutants prior to conventional wastewater treatment. Urine diversion allows for targeted pharmaceutical removal because approximately 64% of pharmaceuticals used by humans are excreted in urine as the active ingredient (Lienert, Burki, & Escher, 2007). Concern for pharmaceutical pollution has been increasingly covered by the U.S. media, with a major focus being on their effects on "public health and harm to the environment" (Blair, Zimny-Schmitt, & Rudd, 2017). Novel technologies, such as ion exchange and biochar adsorption, and advanced oxidation, have been proposed as methods for removal of pharmaceuticals in human urine (Landry, Sun, Huang, & Boyer, 2015; Solanki & Boyer, 2017; Zhang, Sun, Boyer, Zhao, & Huang, 2015). As such, urine diversion is a process by which the benefits of water conservation, nutrient recovery, and targeted pharmaceutical removal are possible, but specific hurdles impeding the implementation of these systems exist that need to be studied prior to adoption of the process.

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The first step in the process of urine diversion is the separation and collection of undiluted urine at the source. It is imperative to have seamless and functional collection, as it is the first step in the process (Boyer & Saetta, 2019). It is also imperative to collect undiluted urine, as the addition of dilution water creates conditions that lead to the failure of the collection fixtures (Liu, Wen, Wang, Zhu, & Hu, 2014; K. M. Udert, Larsen, & Gujer, 2003b). However, urine-collecting fixtures, i.e., nonwater urinals and urine diverting toilets, have been shown to malfunction under real operation when precipitation leads to clogging in the fixtures and collection pipes (Abeysuriya, Fam, & Mitchell, 2013; Berndtsson, 2006; Bristow, McClure, & Fisher, 2006). Urine enters the collection system as fresh urine, which is excreted at pH 5–7 (Putnam, 1971). As it comes in contact with the restroom environment, bacterial and free urease enzyme rapidly hydrolyzes the urea molecule to form ammonia and bicarbonate, a chemical reaction known as urea hydrolysis (K. M. Udert, Larsen, Biebow, & Gujer, 2003).

$$(NH_3)_2CO + 3H_2O \xrightarrow{urease} 2NH_4^+ + HCO_3^- + OH^-$$

The addition of ammonia into solution increases the pH from 5–7 to pH 9 and increases the conductivity as charged ions are added to solution (K. M. Udert, Larsen, Biebow, et al., 2003). At pH 9, calcium and magnesium solids, such as struvite and hydroxyapatite, become supersaturated and precipitate. These hard crystals lead to clogging within the collection systems, beginning at the urinals and toilets. Ironically, these crystals are the slow-release fertilizer product recovered during struvite precipitations from urine and are therefore a loss in nutrients that can be recovered with subsequent nutrient recovery technologies (Ronteltap et al., 2007). Nitrogen losses also become apparent as ammonia volatilizes in the collection systems (Rossi, Lienert, & Larsen, 2009). Therefore, inhibiting urea hydrolysis is a manner of maintaining the function of nonwater fixtures and increasing the efficiency of nutrient recovery, i.e., reducing clogging reducing losses of nutrients due to precipitation and ammonia volatilization (Hannah Ray, Saetta, & Boyer, 2017; Saetta & Boyer, 2017). Research has proven two methods for urea hydrolysis inhibition in urine diversion systems: (1) acid addition and (2) base addition to increase or decrease pH above or below the activity limit of the urease enzyme (Hellstrom, Johannson, & Grennberg, 1999; Jonsson & Vinneras, 2007; Dyllon G. Randall, Krähenbühl, Köpping, Larsen, & Udert, 2016; Hannah Ray et al., 2017; Saetta & Boyer, 2017; Senecal & Vinnerås, 2017). Past research by Saetta and Boyer and Ray et al. has used acetic acid addition because it does not cause any additional precipitation that could harm the collection system (Hannah Ray et al., 2017; Saetta & Boyer, 2017). Saetta and Boyer were able to show that the viability of acid addition at the urinal with the proof-of-concept study published in 2017. Conversely, calcium hydroxide addition has been shown to successfully inhibit urea hydrolysis with an added benefit of precipitating calcium phosphate in collection tanks (Dyllon G. Randall et al., 2016). However, if added at the urinal or toilet, as proposed with acetic addition in this dissertation, precipitation in the collection system would hinder the effectiveness of the inhibition chemical. Hence, acid addition needs to be explored as a way to increase the function of nonwater urinals for continued water conservation and future implementation of urine diversion system.

While the opportunities and drivers exist for urine diversion, a gap exists between research and implementation of the process (see Figure 1-1). This PhD dissertation will propose use the use of cyber-enabled technology as a bridge between the operation and the function of the system in an effort to increase the adoption of urine diversion systems. Cyber-physical systems (CPSs) are the integration of cyber-enabled technologies with physical systems, which are connected via communication technologies (Baheti & Gill, 2011). A CPS designed to control urea hydrolysis in urine diversion systems could be a tool by which the opportunities of urine diversion are actualized at the building-scale. Additionally, a CPS can collect data needed to create data driven models for control of urine diversion systems. Eggimann et al. proposes that real time control and model predictive control has the ability to awaken existing infrastructure by augmenting systems that have been viewed as passive components of our aging infrastructure (Eggimann et al., 2017). A horizon scan of urban wastewater management found that resource recovery, decentralization, and real-time models were among the top 10 most important topics in the field (Blumensaat et al., 2019). This dissertation proposes that data driven models can be used to enlighten conditions within the built environment that can be used to control urine diversion systems and water system within CI buildings.



Figure 1-1. The benefits and challenges of urine diversion system implementation identified in the literature and previous research. The system components are proposed to ensure functionality within urine diversion systems.

Thus, the implementation and adoption of urine diversion systems in commercial and institutional buildings is highly dependent on the functionality of the collection system. The goal of this dissertation was to advance the knowledge of urea hydrolysis process and its inhibition so as to (1) maintain and expand the use of nonwater urinals for water conservation in conventional building plumbing and (2) advance the implementation of urine diversion in the United States. The specific objectives of this research will be answered with the following questions:

1. Can a cyber-physical system monitor and control urea hydrolysis in nonwater urinals?

2. How can urea hydrolysis be monitored and controlled in real time in commercial and institutional buildings?

3. How does urine chemistry and microbiology change as real urine is treated with acetic acid for urea hydrolysis inhibition?

4. How can data driven models advance the implementation of novel water

and wastewater systems?

CHAPTER 2

WATER AND WASTEWATER BUILDING CPS: CREATION OF CYBER-PHYSICAL WASTEWATER COLLECTION SYSTEM CENTERED ON URINE DIVERSION

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Abstract

Decentralized treatment of wastewater has been identified as an area of growth for cyber-enabled sensing and control. One such system that would benefit from embedded cyber components is urine diversion. This research sought to create a cyber-physical system for wastewater collection and treatment. Two subsystems were integrated into the CPS: sensing and actuation. Real-time sensing using low-cost pH and conductivity sensors was used to monitor urine chemistry. Actuation was used to deliver urine to the system and to control urine chemistry. Once integrated, the system used the sensing data to determine when to actuate urine chemistry control pumps. By using urine diversion as a test case, two demonstrations were able to show the applicability of the system created. The first demonstration was able to characterize the flow characteristics of the physical system. The second demonstration was able to mimic and inhibit urea hydrolysis under realistic conditions. Ultimately, the use of this novel CPS approach was able to confirm the benefits of sensing and actuation in wastewater collection and treatment within buildings.

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Introduction

"Cyber-physical systems (CPS) are integrations of computation, networking, and physical processes. Embedded computers and networks monitor and control the physical processes, with feedback loops where physical processes affect computations and vice versa,"(Lee, 2008) with application domains in: communication, security, energy, infrastructure, health care, manufacturing, military robotics, and transportation (Asare, Broman, Lee, Torngren, & Sunder, 2012; Baheti & Gill, 2011; Ponsard, Dallons, & Massonet, 2016; Volkan, Steffen, Tony, & Frank, 2014). A particular focus of CPS is cities and the movement toward "smart" cities. For example, sensor networks can be deployed throughout cities to monitor air quality and inform residents of unsafe levels of pollutants and appropriate level of outdoor activity (Bacco, Delmastro, Ferro, & Gotta, 2017). Underrepresented in CPS research is application to drinking water and wastewater systems. For instance, an article on the applications of water CPS for water sustainability highlights source water quality monitoring and water distribution system monitoring, and mentions control technologies as an area of future research (Z. Wang et al., 2015). Although monitoring water flow and water composition can provide useful information about the system, control is needed to make the system "smart" for smart cities applications.

Water and wastewater systems are poised to leverage the advances in electronics and computer science that is exemplified by CPS. For example, Eggimann et al. discuss how a CPS approach could transform urban water management where sensing and datadriven modeling could be used to monitor and control weather, flooding, green infrastructure, wastewater collection and treatment, and decentralized treatment (Eggimann et al., 2017). However, just like the article on CPS applications to water sustainability (Z. Wang et al., 2015), the paper by Eggimann et al. is mostly at the idea stage and lacking experimental testing and verification. Finally, Blumensaat et al. conducted a horizon scan of urban water management to identify future opportunities and threats many of which centered on data—emerging data, interaction of data and stakeholders, and data-driven modeling (Blumensaat et al., 2019). Again, the horizon scan was focused on the idea stage and not demonstrations of CPS for water management.

One area that Eggimann et al. identify as an area of growth for CPS applications is decentralized wastewater treatment. Decentralized treatment allows for separation of valuable waste streams at the source of production. One such example is urine diversion, whereby urine is passively separated from wastewater at the point of collection by using nonwater urinals or urine-diverting toilets and separate collection pipes from those conveying wastewater to a wastewater treatment plant (Larsen & Gujer, 1996). Urine is stored and treated to recover beneficial nutrient products that would otherwise be produced in highly intensive resource and energy processes. Urine diversion systems (i.e., collecting urine without flush water) also contribute to water conservation. However, due to a naturally occurring reaction called urea hydrolysis, the nitrogen and phosphorus in urine are unstable as urine enters a collection system (K. M. Udert, Larsen, Biebow, et al., 2003). Urea hydrolysis transforms the urea-nitrogen into ammonia and bicarbonate, which raise the pH in urine from 6 to 9. At pH 9, phosphorus-containing minerals are more likely to precipitate on the restroom fixtures and pipe networks (K. M. Udert, Larsen, Biebow, et al., 2003; K. M. Udert, Larsen, & Gujer, 2003a). This loss of

nutrients in the collection system adversely affects the nutrients that can be recovered in subsequent technologies, which are seeking to recover the greatest amount of valuable product. Controlling urea hydrolysis has been an active research topic, with literature pointing to the addition of acid or base as a strategy for inhibiting the reaction (Boyer & Saetta, 2019; Hellstrom et al., 1999; Dyllon G. Randall et al., 2016; Saetta & Boyer, 2017; Simha, Senecal, Nordin, Lalander, & Vinnerås, 2018). As such, a cyber-physical system could be of great benefit for urine diversion systems as an automated, cyberenable method for controlling the addition of inhibiting chemicals.

The goal of this research was to create a wastewater subcomponent of a building water and wastewater cyber-physical system using urine diversion as a test case. The specific objectives were to (1) design cyber-physical system (CPS) to meet wastewater collection criteria, (2) construct and implement CPS, (3) evaluate accuracy of CPS, (4) demonstrate operation and application of the CPS, and (5) identify the implications of the CPS on wastewater systems within buildings. The objectives were accomplished by integrating sensors, controllers, actuators, data storage, and data analysis with nonwater urinals and wastewater piping.

System Description

Biodesign C is a laboratory building on ASU's Tempe Campus. It was constructed to LEED Platinum status and it was opened in May 2018. The building is classified under the commercial and institutional (CI) building construction category. CI buildings are defined as those that are used for healthcare, education, recreation, public works, office space, warehousing, or retail. In CI buildings, water use and wastewater production takes place in the restrooms, cooling and heating, and landscaping (US Environmental Protection Agency, 2012). A CPS was created for water and wastewater monitoring and control within the building with the premise that building occupancy impacts the water use, water quality, and water conservation within the building. Research has shown that water quality can change in premise plumbing, with increased water age in green buildings being a factor towards decreased water quality (William J. Rhoads, Pruden, & Edwards, 2016). Decreasing water use inside the building from water conservation increases the water age within the premise plumbing, as less water is flowing through pipes to meet indoor water demand. Low-flow water fixtures, such as faucets, toilets, and urinals, reduce water use and are features included in LEED buildings for water conservation. Yet, the sizing of premise plumbing remains virtually unchanged from those found in conventional buildings. The decreased flow within the pipes, due to water conservation, and the conventionally sized pipes leads to increased water age and reduced water quality (W. J. Rhoads, Pearce, Pruden, & Edwards, 2015).



Figure 2-1. Water and wastewater building CPS for real-time water quality sensing and increased nonwater urinal functionality for water conservation.

This water and wastewater building CPS seeks to study the changes in water chemistry as a function of time and space (see Figure 2-1). With the use of water quality sensors, water quality can be monitored and controlled depending on floor-specific data. The building has been outfitted with a drinking water CPS, which includes pH, conductivity, temperature, chlorine, dissolved oxygen, and oxidation-reduction potential (ORP) sensors at every breakroom sink. As stated above, the drinking water system is highly influenced by water consumption. In the literature, systems have been described that monitoring water quality in the environment and at specific locations, such as within buildings or at points throughout the distribution system. However, a major component of a CPS are the actions taken from the feedback on the system (Baheti & Gill, 2011) and a gap exists in the literature when it comes to including control in environmental monitoring. As such, the subcomponent on the CPS monitoring the drinking water within the building will fill the gap by controlling water quality via actuating valves. The specific design and operation of the drinking water CPS will be described in an upcoming research paper. The second subcomponent of the CPS described in this research paper seeks to monitor and control urea hydrolysis in nonwater urinals using urine chemistry sensing so as to keep their functionality as a method for water conservation. Nonwater urinals are a common addition to green buildings because of their ability to conserve large amounts of water. Nonwater urinals are also used in urine diversion systems because they are able to collect urine without diluting urine with flush water. However, past implementation has caused to unforeseen difficulties in management of nonwater urinals (Abeysuriya et al., 2013; Blume & Winker, 2011; Bristow et al., 2006). Solutions for increased functionality of nonwater urinals are needed to keep nonwater urinals as

viable options for water conservation in commercial and institutional buildings and ultimately paper of a future urine diversion system (Boyer & Saetta, 2019).

Experimental Methods

A. Physical Components

The urinal testbed held three Kohler Standard Waterless Urinals and three urine storage tanks (see Figure 2-2). The urinals were piped with cast iron to mimic the way urinals are piped in restrooms in CI buildings. Two P-traps were added to the pipe runs to allow for two sampling locations as urine traveled from the urinal to the storage tanks. Each urinal had three sampling locations: trap 1, trap 2, and the storage tank. Three solutions were delivered to the urinal testbed to mimic urine diversion systems: (1) synthetic fresh urine, (2) urease solution, and (3) acetic acid to the urinals. The synthetic fresh urine was made following the recipe presented in Saetta & Boyer, 2017 (Saetta & Boyer, 2017). Its major components include 500 mM as N of urea, 20 mM of phosphate, 4 mM of calcium, and 4 mM of magnesium. The urease solution was made by dissolving 12.64 g of jack bean urease (EC 3.5.1.5, Sigma Aldrich, powder, ~1 U/mg) in 250 mL of deionized water (DI). The acetic acid solution was made by diluting glacial acetic acid (CH3COOH, Fisher Chemicals) to make a 2500 meq/L solution.

B. Cyber Components

Sensing of the urine chemistry in the urinal testbed was conducted using low-cost sensors and microcomputers. Each urinal used three pH and conductivity sensors from Atlas Scientific that were connected to a VizLore U-IoE controller, which holds a Raspberry Pi 3 Model B. The three urinal controllers were in communication with the cloud, which was responsible to listen to telemetry events from the device, i.e., receive payload and send command to devices from cloud. Google cloud platform was used for data analysis and data storage. The Cloud Datastore is a Not Only Structured Query Language (NOSQL) database where the payload from the controllers from each experiment was stored. NOSQL is a key value pair store, which can be easily extended to add more attributes. This helps in using the same database after adding more sensors to the system to add capabilities to the urinal testbed as sensors are developed. The Cloud App Engine was used to host the website and to see the datastream during the experiments. The controllers were connected to the Internet using an Ethernet connection because of poor Wi-Fi connection in the laboratory. Cloud IoT Core was used to authenticate and ingest the datastream from the controllers. Cloud IoT Core helps by adding more devices and ingesting data from devices in a scalable manner. Each controller was authenticated by a using a public private key pair. The controller was connected to Cloud IoT Core via Message Queuing Telemetry Transport (MQTT). MQTT was preferred because of lower bandwidth usage, lower latency, higher throughput and continuous device connection. MQTT also guarantees a Quality of Service 1, which is that the message is delivered at least once. This leads to no data loss. Also, a device connection status is maintained which helps in knowing whether the device is online or offline. Cloud IoT Core uses Pub/Sub for the datastream. The datastream was pushed to an endpoint where the data format was held and then stored the data in Cloud Datastore. Another pull on the datastream was done to show the data visualization on the website. The controller was controlled from the website via Cloud IoT Core Representational State Transfer Application Programming Interfaces (REST APIs). REST API helps in transferring data/command from the website to the cloud. The cloud further interprets the message and send the appropriate command to the device(s). This also helps in exposing different levels of device control to different users in a secure way.



Figure 2-2. Front and back of urinal testbed. Pumps were used to deliver synthetic urine, urease solution, and acetic acid to the three urinals. The sensors were placed inside the two P-traps and in the urine storage tanks.



Figure 2-3. Urinal testbed cyber-physical system schematic showing two subsystems of sensing and actuation.

Three peristaltic pumps were used as actuators on the system. To control the synthetic urine pumps a Raspberry Pi 3 was used as the controller running a custom python program. A cable assembly was created to connect the DB-25 connection on the pump to the General Purpose Input Output (GPIO) outputs on the Raspberry Pi 3, via this connection the pump could be started, stopped, and the pumping direction reversed.

In the cloud, two control logics were used to determine when actuation would occur for one of pumps. The first control logic was a reactive control logic that would activate the pump once a threshold pH > 7 was reached for 4 consecutive measurements by one of the sensors in the first P-trap. The second control logic was a predictive model control logic. Four lasso regression models were used to predict the future 4 pH measurements in the first P-trap. The lasso regression models used the last 20 pH measurements, the last 20 conductivity measurements, the time since the last urination event, and the volume of the last urination event to predict the following pH measurement. The logics were held on the urinal controllers, and data was analyzed based on the logic in use locally. Once the measurements and data met the requirements for the logics, a message was sent from the urinal controllers to the pump controller, and the pumps were actuated accordingly.

Results and Discussion

A. Design, Construction, and Implementation of the CPS

The CPS was designed to monitor and control urea hydrolysis in nonwater urinal systems. A urinal testbed was built as a representative for nonwater urinal systems in commercial and institutional buildings. The cyber components were overlaid on the urinal testbed to create the CPS (see Figure 2-3). The first component of the CPS that was designed was the actuation control. Actuation on the system is an integral component of a CPS. Without actuation, the system cannot respond to the physical components that it is monitoring. The CPS designed in this study was able to control the urine chemistry by way of activating and pausing the system pumps. The pumps were used to mimic the use of nonwater urinals by delivering urine and urease at average volumes. Ultimately, the pump system was designed to have three functions: (1) constant inputs at constant frequencies, (2) constant inputs at random frequencies, and (3) random inputs at random frequencies. The urine and urease delivery pumps acted as a simulator during the creation of the control CPS. The variability found in real restrooms would have introduced too many variables, and would not have provided the data needed to model the system. Therefore, the urine and urease pumps were implemented while the CPS was created and tested, but the pumps for urine and urease are not components of implementation of the control CPS once the system is applied in the real restrooms. By implementing this simulation of urination events into the urinal testbed, the testbed became a tool that could be used to test a myriad of treatments and scenarios for urine diversion research.

The pump controller was able to control up to 3 pumps at a time and in its original iteration could start, stop, or reverse direction on any individual pump. To run an automated pumping experiment the program accepted three input values; "On Time", "Off Time", and "Cycles". The "On Time" variable determined the pump run time, it would remain off for a period determined by the "Off Time" variable in seconds and would repeat for the set number of "Cycles." The second iteration of the pumping program added a random parameter that would make the pumping events happen at random intervals. The urination events would occur at random intervals between 10–20 min and the urination event duration would also be a random variable between 10–40 s for the input number of cycles. There would be a 20 s urease addition event immediately following each urination event.



Figure 2-4. Architecture of the nonwater urinal CPS.

The second component of the CPS was the ability to monitor urine chemistry. Two measurements, pH and conductivity, have been shown to acts as surrogates for the urea hydrolysis reaction when ammonia and urea cannot be directly measured (Hannah Ray et al., 2017). In order to control urea hydrolysis, the CPS had to have the capability of measuring pH and conductivity in real-time. The collection pipes for each urinal were designed to have two P-traps that would hold urine between urination events. The pH and conductivity sensors were placed in the P-traps as a way of monitoring the urine chemistry as a function of time and space. The storage tank for each urinal also held a set of pH and conductivity sensors as a means of monitoring the urine chemistry as urination events occur. When experiments began, the cloud sent a universally unique identifier (UUID) with the start message to all the 3 controllers. This UUID was used to give each experiment a unique fingerprint. Table 2-1 shows the attributes sent by each controller in each payload and Figure 2-4 shows the final architecture of the CPS.

For the third iteration of the program, a publish-subscribe method was used to listen for a message informing the system that pH had risen above the specified set point. When this message was received a third, independent from the other two, pumping event would be initialized for 20 s to add acetic acid to the system. The system would then ignore messages for 10 min and then would return to listening for the message again after that time period elapsed. The 10 min cool down period was used as a buffer to prevent acid addition overdose, because there was a delay in pH readings as they only change after a urination event occurs and moves new liquid into the p-traps where the sensors are located. If an acid addition event occurs just after a urination event ends, it could be up to 20 min until the next urination event occurs. After the cool down period the program would continue to monitor for the acid addition flags until the number of cycles were completed for the urine and urease additions. Details on experimental results using the third iteration of the system can be found in Saetta et al, 2019 (Saetta et al., 2019a). The system design was compared to systems described in the literature and it can be seen that this design fills the gap that exists between the implemented environmental monitoring systems and those proposed in papers of the future applications (see Table 2-2). The vast majority of papers were focused on monitoring environmental systems with sensor networks and data acquisition. While it allows for better understanding of the systems, the monitoring alone does not allow for control of the systems as a CPS proposes. As the popularity of CPS implementation has grown in energy and air handling systems, the water and wastewater field has been slow to implement the control mechanisms in conjunction with monitoring systems. Only five papers proposed the use of monitoring and control in environmental applications. The research presented in this paper is the first application of CPS in urine diversion systems and can be used as a prototype for wastewater monitoring and control within CI buildings.

Attribute	Description
Controller_id	<string> Unique identifier to identify the controller</string>
UUID	<string> Unique identifier to identify the experiment number</string>
datetime	<datetime> Datetime when the measurement was taken on controller</datetime>
c_data1	<float> Value of conductivity Sensor 1 (in µS/cm)</float>
c_data2	<float> Value of conductivity Sensor 2 (in µS/cm)</float>
c_data3	<float> Value of conductivity Sensor 3 (in µS/cm)</float>
ph_data1	<float> Value of pH Sensor 1</float>
ph_data2	<float> Value of pH Sensor 2</float>
ph_data3	<float> Value of pH Sensor 3</float>

Table 2-1. Attributes sent to the cloud with each payload of data
B. Accuracy of the CPS

Data received from the sensors and controllers was compared to data measured with benchtop pH and conductivity meters. The sensors were placed in a beaker to monitor the pH and conductivity of urine undergoing urea hydrolysis. During urea hydrolysis, the urea in urine is hydrolyzed to form ammonia and bicarbonate. The pH of the solution increases from 6 to 9 as ammonia is formed and the conductivity increases as more charged ions are created (urea is an uncharged organic compound). The reaction is catalyzed by the urease enzyme, which was supplied to the urine at t = 0 and at t = 1000 min. The reaction was monitored for 22 h and measurements were taken every 1 min. Fig. 2-2 shows the results for pH and conductivity measured by the 9 CPS sensors and the benchtop analogs. As seen, pH measured by the CPS sensors was less than 1% different from the pH measured by the benchtop meter, showing agreement between a known measurement and the CPS sensors (i.e., pH). On the contrary, the CPS sensor measurements for conductivity were not in alignment with the measurements taken by the benchtop conductivity meter. All 9 CPS conductivity sensors were in agreement with each other, while the measurements by the benchtop conductivity meter were offset by a constant value. This may be due to temperature compensation in the benchtop meters that is not incorporated in the CPS sensors. However, the goal of this project was to monitor the changes in urine chemistry over time. The CPS sensors were able to observe the trend of increasing conductivity over time.

			Monitoring	
Reference	Reference Year		location	Limitations
			location	
(Hart & Martinez,	2006	Environmental Sensor	Natural	Proposed
2006)		Networks: A revolution in the environment		application
		earth system science?		
(Eggimann et al.,	2017	The Potential of Knowing	Water and	Proposed
2017)		More: A Review of Data- wastewater a		application
		Driven Urban Water		
		Management		
(Z. Wang et al.,	2017	Cyber-Physical Systems for	Water	Proposed
2015)		Water Sustainability:	environment	application
		Challenges and Opportunities		
(Boyer & Saetta,	2019	Opportunities for Building-	Urine diversion	Proposed
2019)		Scale Urine Diversion and		application
		Challenges for Implementation		
(Blumensaat et al.,	2019	How Urban Storm- and	Water and	Proposed
2019)		Wastewater Management	agement wastewater	
		Prepares for Emerging		
		Opportunities and Threats:		
		Digital Transformation,		
		Ubiquitous Sensing, New Data		
		Sources, and Beyond - A		
		Horizon Scan		

Table 2-2. Literature related to environmental monitoring and proposed cps use for water or wastewater management

(Pule, Yahya, &	2017	Wireless sensor network: A Water quality		Monitoring	
Chuma, 2017)		survey on monitoring water		review	
		quality			
(Kelly,	2013	Towards the Implementation of	Hot water, power	Monitoring	
Suryadevara, &		IoT for Environmental consumption			
Mukhopadhyay,		Condition Monitoring in			
2013)		Homes			
(Wu, Kong, &	2013	Water Environment Monitoring	Water quality Monitoring		
Zhang, 2013)		System Based On ZigBee			
		Wireless Sensor Network			
(Lambrou,	2014	A Low-Cost Sensor Network	Water quality	Monitoring	
Anastasiou,		for Real-Time Monitoring and			
Panayiotou, &		Contamination Detection in			
Polycarpou, 2014)		Drinking Water Distribution			
		Systems			
(Cloete, Malekian,	2016	Design of Smart Sensors for	Water quality	Monitoring	
& Nair, 2016)		Real-Time Water Quality			
		Monitoring			
(Bacco et al.,	2017	Environmental Monitoring for	Air quality,	Monitoring	
2017)		Smart Cities ambient			
			conditions, traffic		
(Csáji, Kemény,	2017	Wireless Multi-Sensor	Air quality,	Monitoring	
Pedone, Kuti, &		Networks for Smart Cities: A	ambient		
Váncza, 2017)		Prototype System With	conditions		
		Statistical Data Analysis			

(Dong, Meyland,	2017	A Case Study of an Water quality Monitor		Monitoring
& Karaomeroglu,		Autonomous Wireless Sensor		
2018)		Network System for		
		Environmental Data Collection		
(Bhardwaj, Gupta,	2018	Towards a cyber-physical era:	Water quality	Monitoring
& Gupta, 2018)		soft computing framework		
		based multi-sensor array for		
		water quality monitoring		
(Y. Chen & Han,	2018	Water quality monitoring in	Water quality	Monitoring
2018)		smart city: A pilot project		

The reaction was monitored for 22 h and measurements were taken every 1 min. Fig. 2-2 shows the results for pH and conductivity measured by the 9 CPS sensors and the benchtop analogs. As seen, pH measured by the CPS sensors was less than 1% different from the pH measured by the benchtop meter, showing agreement between a known measurement and the CPS sensors (i.e., pH). On the contrary, the CPS sensor measurements for conductivity were not in alignment with the measurements taken by the benchtop conductivity meter. All 9 CPS conductivity sensors were in agreement with each other, while the measurements by the benchtop conductivity meter were offset by a constant value. This may be due to temperature compensation in the benchtop meters that is not incorporated in the CPS sensors. However, the goal of this project was to monitor the changes in urine chemistry over time. The CPS sensors were able to observe the trend of increasing conductivity over time.



Figure 2-5. pH and conductivity results comparing CPS sensors to benchtop meter measurements. Each CPS sensors is denoted as the PXCY or CXCY where X corresponds to the sensor number and Y corresponds to the controller number. There were 3 sensors per controller.



Figure 2-6. Conductivity measurements in Trap 1 for the three urinals on the urinal testbed. Tracer tests were run for urination events every (a) 3 min, (b) 10 min, and (c) 30 min. A sodium chloride solution was pumped into the urinals after 20, 5, and 3 urination events, respectively.

C. Demonstration and application of the CPS

Once the CPS sensor accuracy was established, the CPS was used in two demonstration experiments to illustrate the range of capabilities and new insights that could be gained with a CPS of this design. The first demonstration experiment was used to characterize the flow in the urinal testbed as plug flow reactor (PFR) versus completely mixed flow reactor (CMFR). A PFR is an ideal reactor type where there is no mixing in the direction of flow (Howe, Hand, Crittenden, Trussell, & Tchobanoglous, 2012). A CMFR is a second ideal reactor type that has uniform conditions within the reactor, as reactants instantaneously mixes with the contents within the reactor (Howe et al., 2012). In ideal conditions, a tracer test on PFR shows a spike in concentrations that is sharp and fast moving through the systems, which returns to background levels after the "plug" of fluid has gone through the reactor. In a CMFR, the tracer will have more mixing, which elongates tracer curve, showing an increasing in the amount of time it takes the tracer to exit the reactor and for the system to go back to background levels. This research allows for a feasible solution to study the flow patterns within wastewater plumbing systems, as there has never been implementation of sensors within wastewater plumbing for this purpose. Figure 2-5 shows the results of conductivity as a sodium chloride solution was pumped into the testbed. With that data, the mean residence time can be calculated. The mean residence time was compared to the theoretical hydraulic residence time (HRT, $\tau = V/Q$), as seen in Figure 2-7a.

It can be seen that the theoretical HRT overestimated the time it takes the pulse to travel through the system for the 10 min and 30 min frequencies, while it underestimates the time for the 3 min frequency. According to Howe, et al, the mean residence time is "the

average amount of time that water stays in the reactor as determined by the tracer test" and that, "ideally, the mean residence time is equal to the hydraulic residence time, but that is generally not the case (Howe et al., 2012)."



Figure 2-7. (a) Calculated mean residence time (colored bars) and theoretical hydraulic residence time (striped bars) for the three urinals at three urinations frequencies. (b) Urea concentrations for first-order rate reactions of urea hydrolysis using calculated mean residence time (colored bars) and theoretical hydraulic residence time (striped bars). Results for modeling the system as a PFR are seen in blue and for a CMFR in orange.

These results are important when it comes to modeling the chemical reactions occurring in the system because using the theoretical HRT instead of the mean residence time will give different results. Figure 2-7b shows the calculations of the urea concentrations in the effluent of PFR and CMFR reactors using the calculated mean residence time and the theoretical HRT. The first-order rate equations were solved using k = 8.0×10^{-6} s⁻¹, the theoretical HRT of the system (V = 1550 mL and Q_{3min} = 79 mL/min, Q_{10min} = 23.7 mL/min, and Q_{30min} = 7.9 mL/min), and the mean residence time calculated from the tracer test. The differences between the concentrations calculated with the

theoretical HRT and the mean residence time increase as the frequency of urinations increases, with the 30 min urination frequency showing the largest difference between the theoretical HRT and the mean residence time concentrations. At lower urination frequencies, i.e., 30 min between urinations, the results show the importance of having an accurate HRT of the system, especially because the modeled urea concentrations can differ by approximately 5%. Based on prior research, it was determined that restrooms with lower use frequency could lead to increased urea hydrolysis, and therefore more malfunctions in the urinals (Saetta et al., 2019a). A second result from Figure 2-7b is the similarity between the PFR and CMFR performance. This is due to the low k value that is characteristic of urea hydrolysis equations and was determined in previous studies (Saetta et al., 2019a).

Prior research into urea hydrolysis in nonwater urinals was not able to show the reactor characteristics that the CPS was able to illuminate. Saetta and Boyer, 2017 concluded that a study on nonwater urinal flow dynamics was necessary to illuminate the reactions that occur as urine travels through collection systems (Saetta & Boyer, 2017).



Figure 2-8. Flow chart of the information the CPS was able to provide and how it combines with previously known information to control the chemical reaction in nonwater urinals.

Using the CPS allows for characterizing of the effects of use frequencies on the reactor dynamics (seen in Figure 2-7), which was then combined with the known reaction kinetics (data from laboratory experiments), to create the needed logics to control the urea hydrolysis reaction in nonwater urinals. Figure 2-8 shows a flow chart of the information attained from the CPS to control urea hydrolysis. Without the CPS, nonwater urinals would remain a black box, where urine chemistry undergoes rapid change without being fully understood. Implementation of the CPS allowed for enhanced data acquisition, as prior experiments were only able to see the reaction occurring at a lower resolution. Saetta and Boyer, 2017 was only able to show measurements taken every 30 min, which was not specific enough to determine the flow characteristics of the nonwater urinals (Saetta & Boyer, 2017).

Building on the results from the tracer test, the second demonstration experiments, as detailed in Saetta, et al, 2019, was to use the CPS to conduct mimicking urea hydrolysis experiments (Saetta et al., 2019a). Urea hydrolysis is the reaction that converts the urea in urine into ammonia and bicarbonate under the presence of the urease enzyme (K. M. Udert, Larsen, Biebow, et al., 2003). As this reaction occurs, pH increased from 6 to 9 due to the formation of ammonia and the conductivity increases as more charged molecules are introduced into the urine matrix (the urea molecule is neutral) (Hannah Ray et al., 2017). The CPS was used to monitor urine pH and conductivity in real time as synthetic urine and jack bean urease were delivered into the urinals at a constant flow and volume for 4 h (see

Figure 9). The real time measurements were able to capture the increase in pH and conductivity as urine rested in the collection pipes between urination events. Previous studies were only able to show the upward overall trend in measurements, as opposed to the gradual increase in the measurements before the measurements decreased as fresher urine entered the pipes (Hannah Ray et al., 2017; Saetta & Boyer, 2017).

The CPS was then used to control urea hydrolysis by three control logics: acid addition after every urination event, reactive pH threshold control logic, and predictive model control logic (Saetta et al., 2019a). Results of the different control logics can be seen in Saetta et al. 2019 (Saetta et al., 2019a). The reactive and predictive control logic experiments were made possible by the use of the sensors and cloud computing. The determination of reaching either control logic actionable point was conducted in the cloud, and the message for actuation was sent to the controllers via the internet. A user interface was created to set up experiments on the urinal testbed. The interface was used to calibrate the sensors and to decide experiment parameters, such as volume of urine additions and frequency of additions. The user interface also updated data visualization graphs in realtime as experiments were conducted. In combination with the CPS, the user interface makes the urinal testbed a tool for future research on urinal operation and design.

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Figure 2-9. Results for demonstration experiment mimicking urea hydrolysis. CPS sensor measurements are shown by the light circles. Accuracy of the CPS conductivity sensors was monitored by spot checks using a benchtop meter, as indicated by the filled circle, diamond, and triangle.

D. Implications and contributions of the CPS

The CPS created in this research project is the first system to monitor and control urea hydrolysis in nonwater urinals. As such, it has the ability to transform wastewater and urine collection within buildings. Acid addition at the urinal increases the functionality of nonwater urinals, by inhibiting the urea hydrolysis reaction and decreasing the possibility of precipitation of minerals that lead to clogging (Saetta et al., 2019a). Therefore, it eliminates malfunctioning due to clogging, and allows for continued water conservation in existing buildings and opportunities for increased water conservation in new buildings. Moreover, this CPS could be implemented within existing infrastructure where nonwater urinals are used to reduce indoor water use, or it could be implemented as novel infrastructure whereby the urine diversion system controls the ratio of urea to ammonia in the collected urine for subsequent resource recovery (Hannah Ray, Perreault, & Boyer, 2019). The specific contributions of the CPS are: (1) improve water conservation in existing buildings by reducing precipitation-induced clogging and malfunction, (2) facilitate the implementation of urine diversion by removing the single point of failure in urine collection, and (3) enable tailored recovery of urea or ammonia in multi-story buildings.

The CPS described in this research was also able to manipulate a reversible enzymatic reaction in real-time by reacting to changes in urine chemistry. Urea hydrolysis can be reversibly inhibited in order to create desired chemical conditions at the point of collection, while allowing for the chemical reaction to progress in the storage tanks to a predetermined final composition (Hannah Ray et al., 2017). The ability to respond to the changes in urine chemistry in real-time make this CPS unique and a system that uses sensing data and actuation to control urea hydrolysis is not currently available. At the scale of multi-story buildings, there has not been any application of CPS technology to control urine chemistry for urine diversion or water conservation, as shown in this study. Applications of urea hydrolysis control at the point of collection have focused on redesign of urinals and toilets, mainly through the use of calcium hydroxide as the inhibiting chemical, where precipitated nutrient products must be manually collected from each individual urinal and toilet (Flanagan & Randall, 2018; Dyllon G. Randall et al., 2016). Saetta et al, 2019 used calcium hydroxide in existing nonwater urinals and found losses of phosphate in the system due to precipitation of calcium phosphate (Saetta et al., 2019a). Therefore, base addition creates unwanted precipitation and would directly impact the two main goals of implementing the CPS (i.e., improving the function of existing nonwater urinals and inhibiting urea hydrolysis).

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Finally, depending on the acid addition frequency and control logic used, the CPS allows for control of nitrogen speciation. If coupled with a urea recovery technology, such as recovery via membrane separation (Hannah Ray et al., 2019), then acid addition would be implemented to prolong urea hydrolysis inhibition necessary to keep urea as the main form of nitrogen. Conversely, if the goal of the system is to recover ammonia, such as recovery via ammonia stripping (Jagtap & Boyer, 2018), then acid addition is only necessary for urea hydrolysis in the urinals and drainpipes and urea hydrolysis can progress in the storage tank.

Conclusions

The cyber-physical system created for this research project was able to illuminate what was previously a black box in urine collection systems. In combination with a drinking water CPS, the water and wastewater building CPS will have the unique opportunity to monitor and control water quality and urine chemistry in real time. The system fills a gap in research that has been identified between environmental monitoring and proposed use of CPS in smart cities. The urine diversion CPS described in this research paper was able to mimic realistic conditions of real restrooms in the lab setting. The CPS was used as a tool to understand the system that had not been studied to this detail prior to this study. Moving forward, the CPS components used here should be applied to real nonwater urinal systems in an effort to find an actuation control that could be implemented without needing to build a CPS for every nonwater urinal in use.

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CHAPTER 3

REAL-TIME MONITORING AND CONTROL OF UREA HYDROLYSIS IN CYBER-ENABLED NONWATER URINAL SYSTEM

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Abstract

This research used a cyber-physical system (CPS) to monitor and control the extent of urea hydrolysis in nonwater urinals. Real-time pH and conductivity data were used to control urea hydrolysis inhibition under realistic restroom conditions with acetic acid addition. Variable urination frequencies and urination volumes were used to compare three conditions that affect the progression of urea hydrolysis. Mechanistic and conceptual models were created to evaluate the factors that influence the progression of urea hydrolysis in nonwater urinals. It was found that low urination volumes at low frequencies created ideal conditions for urea hydrolysis to progress. Alternatively, high urination volumes at high frequencies created pseudo-inhibitory conditions because it did not allow for sufficient reaction time or mixing with older urine in the urinal trap. The CPS was used to control urea hydrolysis inhibition by two logics: (1) reactively responding to a pH threshold and (2) predictively responding to past measurements using four lasso regression models. Results from the control logic experiments showed that acid was added once per hour under low use conditions and once in a 4 h experiment for high use conditions. The CPS allowed for full control of urine chemistry in the nonwater

urinal, reducing the conditions (i.e., clogging and malodor) that have led to the removal of nonwater urinals in the United States.

Introduction

The process of separately collecting urine, known as urine diversion, has been proposed and trialed as an alternative to conventional wastewater treatment because it opens the possibilities of nutrient recovery, pharmaceutical removal, and reduced environmental impact (S. K. Ishii & Boyer, 2015; Larsen & Gujer, 1996; Larsen, Lienert, Joss, & Siegrist, 2004). Urine diversion begins at the restroom fixtures, where urine is diverted from the wastewater collection system using nonwater urinals and urinediverting toilets (UDTs). In the United States, an opportunity exists to implement urine diversion where nonwater urinals are planned for water conservation in new green buildings (American Society of Mechanical Engineers, 2006). According to green building design guidelines, such as Leadership in Energy and Environmental Design (LEED) and Building Research Establishment Environmental Assessment Method (BREEAM), designers are given a choice between high-efficiency urinals or nonwater urinals.(Kubba, 2017) Additionally, LEED's crediting system allots 1 credit to "sustainable wastewater management" implementation, which includes reduction of wastewater production or nutrient recovery (US Green Building Council, 2013). Therefore, when designing green buildings, urine diversion for water conservation and added nutrient recovery benefits could be of value to the building stakeholders (D. G. Randall & Naidoo, 2018). These techniques could be implemented immediately using nonwater urinals since they are commonly used in commercial and industrial (CI) buildings in the United States and extended to UDTs when they become more widely used.

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A concern when implementing urine diversion systems is the possible loss of nitrogen during collection and storage (K. M. Udert, Larsen, & Gujer, 2003b). Nitrogen undergoes rapid transformation from urea in fresh urine to ammonia in hydrolyzed urine due to the presence of the urease enzyme (K. M. Udert, Larsen, Biebow, et al., 2003). This reaction is called urea hydrolysis and it rapidly occurs in urine diversion systems (K. M. Udert, Larsen, Biebow, et al., 2003). Those seeking to recover nitrogen from urine have an interest to inhibit or control urea hydrolysis because of potential losses due to the formation of ammonia (K. M. F. Udert, C.; Münster, M.; Larsen, T.A.; Siegrist, H.; Gujer, W.;, 2003). In the literature, urine stabilization has been shown by two methods: partial nitrification or urease inhibition (Feng, Wu, & Xu, 2008; Hellstrom et al., 1999; Dyllon G. Randall et al., 2016; Hannah Ray et al., 2017; K. M. Udert, Fux, et al., 2003). Biological nitrification has been shown to partially nitrify the ammonia (which was originally present in urine as urea) into nitrate, creating ammonium nitrate as a fertilizer product (Feng et al., 2008; K. M. F. Udert, C.; Münster, M.; Larsen, T.A.; Siegrist, H.; Gujer, W.;, 2003). Conversely, urease inhibition in human urine has only been successful by increasing or decreasing the pH of urine to create conditions where the enzyme is not active and the urea remains unchanged ($pH_{optimal} = 7-8$) (Hellstrom et al., 1999; Dyllon G. Randall et al., 2016; Hannah Ray et al., 2017). Previous research has shown that acetic acid addition inhibited urea hydrolysis when added after every urination event and calcium hydroxide has been shown to inhibit urea hydrolysis when added in the urine storage tanks (Flanagan & Randall, 2018; Dyllon G. Randall et al., 2016; Saetta & Boyer, 2017). However, there is no published research on optimizing the inhibition of urea hydrolysis and stabilization of urea under realistic restroom conditions such as random

frequency of users. In real restrooms, humans interact with the system by varying their use in the following three ways: (1) variable urination volume, (2) variable urination frequency, and (3) natural fluctuations in urine composition (Rossi et al., 2009). An experiment on urine chemistry in nonwater urinals should be able to manipulate these factors in order to be as close as possible to a real restroom. The research presented below varied the urination volume and frequency, making it the closest representation of urea hydrolysis in nonwater urinals in real restroom systems during times of use.

There is an opportunity to control urea hydrolysis conditions in restrooms using a cyber-physical system (CPS). A CPS "[represents] the integration of physical and embedded systems with communication and [information technology (IT)] systems," that range from small to large, city-wide applications (Törngren et al., 2017). Cyber-physical systems in commercial and industrial buildings have been used mostly for energy systems management (Kleissl & Agarwal, 2010; Vasavada & Sasidhar, 2017; S. Wang, Zhang, Shen, & Xie, 2011). The water and wastewater infrastructure sector has been identified as an area of critical infrastructure that can benefit from integration with cyber systems (Baheti & Gill, 2011; Volkan et al., 2014). A CPS would give the ability to monitor and control urea hydrolysis in real-time, which is a novel approach when studying urine diversion systems. Past research has shown that monitoring of pH and conductivity can be used as a surrogate for urea hydrolysis progression (Hannah Ray et al., 2017). Therefore, a CPS equipped with pH and conductivity sensors was built for this study. Using that data, actuators controlling the addition of acid or base can be controlled to deliver chemical inhibitor only when necessary. With such a system, green CI buildings

would be able to continue conserving water, while a CPS aids in maintaining the function of nonwater urinals.

The goal of this research was to use a nonwater urinal cyber-physical system to control urea hydrolysis in multi-story, commercial and institutional (CI) building restrooms under realistic conditions. The specific objectives were to (1) mimic and inhibit urea hydrolysis in nonwater urinals using constant conditions, (2) create a mechanistic and conceptual model for urea hydrolysis in nonwater urinals, (3) compare two control logics for urea hydrolysis inhibition based on low frequency of use conditions, and (4) compare low and high frequency of use conditions using the same control logic. The motivation of controlling urea hydrolysis in these experiments was twofold: (1) to decrease clogging and malfunction of nonwater urinals and collection pipes and to (2) maximize nutrient content in the storage tanks at the end of the experiment for subsequent nutrient recovery. The objectives were accomplished by conducting the experiments with a physical system modeled after a multi-story CI building restroom considering restroom plumbing codes and real-time sensing, with ultimate progression towards a system that included realistic restroom use and urea hydrolysis control using the real-time sensing data.

Materials and Methods

Materials and Urinal Testbed

Synthetic urine and jack bean urease (EC 3.5.1.5, Sigma Aldrich, powder, ~1 U/mg) were used throughout the experiments (recipe in supporting information (SI, Appendix A), Table 3-S1). The synthetic urine recipe was made to contain 500 mM urea as N, 20 mM P (NaH₂PO₄), 4 mM Mg (MgCl₂·H₂O), and 4 mM Ca (CaCl₂·H₂O) and has

been used in previous literature (Landry & Boyer, 2013; Hannah Ray et al., 2017; Saetta & Boyer, 2017). The synthetic fresh urine was adjusted pH 6 using sodium hydroxide prior to each experiment to ensure all experiments began with urine at pH 6. The jack bean urease solution was made by dissolving 12.64 g of jack bean urease in 250 mL of deionized (DI) water. The solution was kept on ice throughout the experiments to avoid a loss in urease activity. Glacial acetic acid (CH₃COOH, Fisher Chemicals) was used to make an acetic acid solution with a concentration of 2500 meq/L. Calcium hydroxide (Ca(OH)₂, Fisher Chemicals) was dissolved in 18.2 M Ω water and stirred continuously overnight to make a 0.64 M Ca(OH)₂ slurry. This concentration was made to equal 5 g Ca(OH)₂ per liter of urine, as documented in the literature (Dyllon G. Randall et al., 2016). It was made the day prior to the experiments and was sealed to the atmosphere while it stirred overnight. All experiments were conducted at a laboratory temperature of 22°C.

The urinal testbed held three Kohler Steward Waterless Urinals. Three peristaltic pumps and tubing were used to deliver the three solutions into each urinal (synthetic urine, jack bean urease solution, and acid or base solution). The outlet of the urinals was piped with 2 in. (5.08 cm) cast iron pipes, which were constructed to have two consecutive P-traps (photos of the urinal testbed in SI). The distance between trap 1 and trap 2 was approximately 25 cm in length. The consecutive P-traps allowed for monitoring of urine chemistry as a function of time and space. The urinal testbed had an approximate residence time of 65 min when operating with constant parameters (see SI for details). The first P-trap also served as a surrogate measurement for urine chemistry closest to the chemistry inside the urinal trap because sensing inside the urinal trap was

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not possible. Conductivity and pH sensors were placed at each P-trap for real-time monitoring and samples were collected from the P-traps at 30 min intervals. Two peristaltic pumps were used for sample collection from the P-traps. The pipes conveyed the urine into 37.9 L linear low-density polyethylene (LLDPE) tanks (Tamco, US Plastic). Each storage tank also held a conductivity and pH sensor. A total of three locations per urinal were monitored in real-time for conductivity and pH throughout the urinal testbed.

Hardware

Each urinal was outfitted with its own controller, which was in communication with a central controller. The controllers consisted of a Raspberry Pi 3 Model B and six sensors. Each urinal controller controlled 3 pH sensors (Atlas Scientific) and 3 conductivity sensors (Atlas Scientific). The three solution pumps were connected a Raspberry Pi, which controlled the length of time each pump was turned on and the frequency at which it delivered its solution to the urinals.

Software

Python 2.7.13 code was used to create programs for the sensors controllers and the pump controller. The data collected with the sensor controllers was uploaded to the cloud via Google Cloud Pub/Sub. Data were stored in a Google Cloud Datastore, where it was archived for future analysis. Measurements at the nine locations within the urinal testbed were taken at 1 min or 15 s intervals depending on the experiment. The sensors were instructed to begin logging measurements at t = 0, even though the urine did not reach all sensors until the testbed traps and tanks were filled.

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	Urination	Urination			
Experiment	Volume,	Frequency,	Addition	Addition frequency	
	mL	min			
Mimicking urea	237	10	_	_	
hydrolysis					
Inhibiting urea hydrolysis	227	10	A patie paid	10 min	
with acetic acid	237	10	Acetic acid	10 11111	
Inhibiting urea hydrolysis	225	10	Calcium		
with calcium hydroxide	237	10	hydroxide	10 min	
Reactive control logic	110 5				
with low urination	110.3 -	10 - 20	Acetic acid	pH 7 threshold	
frequency	4/4				
Predictive control logic	110 5				
with low urination	110.3 -	10 - 20	Acetic acid	Predictive model > pH 7	
frequency	4/4				
Reactive control logic	110 5				
with high urination	118.3 -	1 – 10	Acetic acid	pH 7 threshold	
frequency	474				

Table 3-1. Experiments and parameters used for urination volume, urination frequency, addition used, and addition frequency.

Mimicking and inhibiting urea hydrolysis

Three experiments were conducted to mimic and inhibit urea hydrolysis on the urinal testbed (see Table 3-1). The experimental design was based on experiments presented by Saetta and Boyer, 2017 (Saetta & Boyer, 2017). For the mimicking experiments, synthetic urine (237 mL) and jack bean urease (2.5 mL) solutions were

added to the urinals concurrently for 20 s every 10 min for 4 h (25 urinations per urinal). Conductivity and pH measurements in the P-traps and storage tanks were taken every 1 min. Grab samples for further analysis were drawn from the P-traps every 30 min. For urea hydrolysis inhibition experiments, the experimental design remained unchanged, except for the addition of either acetic acid or Ca(OH)₂ addition (2.5 mL, 20 s) to the urinals immediately following the urine and jack bean urease additions.

Urea hydrolysis control logic and random use frequency experiments

In order to realistically mimic urea hydrolysis in nonwater urinals, a lessprescribed experimental design was used in the following three experiments (see Table 3-1). For the remaining experiments, conductivity and pH measurement frequency was increased to take measurements every 15 s. Two major parameters were changed in the experimental design: (1) urination volume and frequency and (2) acid addition frequency. First, using the pump controller described above, a code for variable urination volume and frequency was included in the remaining experiments. This accounted for randomness in use of real restrooms. The pump controller program was given a range of time to turn on the pumps (urination volume) and a range of time to wait between urination events (urination frequency). Maintaining the urine pump at the same flow rate (711 mL/min), the pump controller logic chose a variable urination time between 10–40 s, which is range that includes a volume half and double the size in the mimicking and inhibiting experiments above (118.5–474 mL of urine per urination event). For urination frequency, two frequency ranges were used in the pump controller logic: (1) low frequency and (2) high frequency. The low frequency logic allowed the controller to

randomly decide the time between urination events between 10–20 min and the high frequency was between 1–10 min.

Two control logics were used to determine the frequency of acid addition as a proof of concept. The first control logic was a reactive control of urine pH in trap 1. After the experiments were started, the sensor controllers were directed to measure pH, while counting the amount of measurements that were above pH 7.0. Once four measurements were above pH 7.0 for urine in trap 1 of any urinal, the sensor controllers sent a message to the pump controller to activate the acid addition pump. Four measurements correspond to 1 min in the experimental time and add up to the frequency of measurements used in the mimicking urea hydrolysis experiment (measurements every 1 min). This was chosen to ensure that pH was constantly above pH 7 and not fluctuating below and above pH 7. The threshold of pH 7 was chosen as the limit in order to allow the urea hydrolysis reaction to increase the pH of fresh urine from 6 to 7, which is one magnitude increase in H^+ concentration. Acid additions were kept constant to be 20 s in length, which was equal to 2.5 mL of acid added. The second control logic was a predictive control of urine pH in trap 1. Four lasso regression models were made with the data collected in the reactive control experiments that would predict the future four steps in pH measurements, using the previous 20 pH measurements, 20 conductivity measurements, the length of time since the last urination event, and the urination time of the previous urination event (lasso regression models in SI). The R^2 values for the four models were as followed: 0.99, 0.97, 0.95, and 0.94. The root mean square errors for the four models were as followed: 0.02, 0.04, 0.05, and 0.06. Once all four models (meaning the next 4 pH steps) predicted a pH

above 7.0, the sensor controllers sent a message to the pump controller to activate the acid addition pump.

Three experiments were conducted using a combination of the realistic experimental parameters (see Table 3-1). The first experiment used the low-use frequency and the reactive pH control. The second experiment used the low-use frequency and the predictive pH control. Lastly, the third experiment used the high-use frequency and the reactive pH control. These experiments will be compared for their ability to inhibit urea hydrolysis. This study did not optimize the pH threshold, volume of acid added per event, or the control logics. Future research is needed to further optimize the methods used in this research for urea hydrolysis inhibition.

Analytical methods

The pH and conductivity of urine in the testbed was measured using the controllers described in the sections above. Grab samples were taken using a peristaltic pump from trap 1 and trap 2 of the testbed every 30 min for further chemical analysis. Samples were only taken when urine was present in the traps, therefore the t = 30 min sample was not taken for experiments with low urination frequency. Samples were immediately acidified with sulfuric acid to pH < 2. All samples were filtered prior to analysis using a 0.2 µm nylon syringe filters (Environmental Express). Samples were diluted with acidified 18.2 M Ω water with a 150× dilution factor to fall within the calibration curves. Ammonia and phosphate were measured using a Lachat QuikChem 8500 Series 2 Flow Injection Analysis system. Ammonia was measure following the EPA NPDES Method 350.1 phenolate method. Phosphate was measured following the EPA NPDES Method 365.1 molybdate based method. Cations (sodium, potassium, calcium, and magnesium) were

measured using a Thermo Scientific Dionex ICS-5000+ with a Dionex IonPac CS12A column.

Mechanistic and conceptual models of urea hydrolysis inhibition control

A mechanistic, first-order rate model was made to evaluate factors that influence urea hydrolysis in the urinal testbed in the urinal trap and trap 1. Urea hydrolysis has been shown to follow first-order reaction in published literature (Connolly, Jackson, Rothman, Klapper, & Gerlach, 2015; Singh, Singh, & Singh, 1992). The model predicted the concentration of ammonia as a function of time using the volume of urination, volume of urine remaining from previous urination event, and experimental time. The model parameters can be seen in Table 3-S4. The model assumed that the urine in the trap is displaced in a plug flow manner as new urine enters the urinal trap, then completely mixes to form a new concentration in the urinal trap before reacting for 10 min (see Fig. 3-S5). The resulting first-order decay of urea was used to find the first-order rate constant for the conditions tested, including urease activity. The first-order rate constant was found to be 8.0×10^{-6} s⁻¹, which is of same order of magnitude to values found in the literature $(2.9 \times 10^{-6} - 2.6 \times 10^{-5} \text{ s}^{-1})$ (Singh et al., 1992; Yadav, Kumar, Singh, & Relan, 1987). A sensitivity analysis was conducted on the k-constant value and results are shown in the SI. Insights from the mechanistic model were used to create a causal loop diagram (CLD) as a conceptual model for the effects of urination frequency and volume on urea hydrolysis in order to understand the external forces, i.e., human interactions and randomness that are exerted on the system (Wolstenholme, 1999).

Results and Discussion

Mimic and inhibit urea hydrolysis

The first objective of this research was to mimic and inhibit urea hydrolysis using the urinal testbed CPS. Urea hydrolysis was mimicked using full-scale urinals in previous research (Saetta & Boyer, 2017). Previous research has also shown that synthetic urine and jack bean urease were able to mimic urea hydrolysis in real urine (Hannah Ray et al., 2017; Saetta & Boyer, 2017). However, this study sought to completely characterize the urea hydrolysis process in the urinals, through the drain pipes, and in the storage tanks. The CPS allowed for real-time measurements of pH and conductivity, with a resolution of 1 measurement per min. Fig. 3-1 shows the pH in trap 1 for three experiments that were conducted, as these measurements are the closest to pH inside the nonwater urinal trap. It is an improvement from the data collected in Saetta and Boyer, 2017, where pH and conductivity of urea hydrolysis in nonwater urinals was measured every 30 min. The pH results show that urea hydrolysis was successfully mimicked in the nonwater urinals, as expected. The pH increased from pH 6 to pH above 8 in 2 hours. With this higher resolution, it is interesting to see the effects of new urine being added every 10 min, as seen in the step changes in pH every 10 min after urination events occur. While the urine sits in the trap, the pH increases with a constant slope, before fresh urine replaces the hydrolyzed urine out of the trap. Subsequent modeling was used to understand the effects of mixing on urea hydrolysis in the urinal trap. pH and conductivity results for trap 2 and the storage tank can be seen in Fig. 3-S1.

Two chemicals were used for the urea hydrolysis inhibition experiments to ensure that a pH above and below the optimal pH of urease was created (pH_{optimal} 7–8)

(Krajewska, 2009). Acetic acid was used in previous work to inhibit urea hydrolysis by decreasing the pH of urine to below the optimal pH for the urease enzyme (Hellstrom et al., 1999; Hannah Ray et al., 2017; Saetta & Boyer, 2017).



Figure 3-1. Trap 1 synthetic urine pH and conductivity for (a) mimicking urea hydrolysis, (b) inhibiting urea hydrolysis with acetic acid, and (c) inhibiting urea hydrolysis with calcium hydroxide experiments. Data is shown for each urinal. The solid lines correspond to the values at t = 0 of the synthetic urine. Data points were taken every 1 min. Data for urinal 1 in (b) is not shown due to technical difficulties with the sensor controller.

Calcium hydroxide has been used as an inhibitor by increasing the pH to above 12.5 (Flanagan & Randall, 2018; Dyllon G. Randall et al., 2016). During each inhibition experiment, inhibition chemicals were added after every urination event. The pH for the two inhibition experiments show that the pH was decreased and increased to the expected pH, where it then stabilized for the entirety of the experiment (Fig. 3-1). pH and conductivity results for trap 2 and the storage tank can be seen in Fig. 3-S2 and 3-S3 for acetic acid and calcium hydroxide addition, respectively.

In order to confirm if urea hydrolysis was occurring, grab samples were taken from traps 1 and 2 every 30 min. Results for ammonia, phosphate, calcium, magnesium, sodium, and potassium are shown in Fig. 3-2. Phosphate and calcium were measured by Saetta and Boyer; hence the additional measurements shown here provide a better insight into the inhibition capabilities of these chemicals (Saetta & Boyer, 2017). To begin with, ammonia is a direct product of the urea hydrolysis reaction, so urea hydrolysis inhibition can be confirmed if ammonia is not produced. Fig. 3-2 shows the average concentrations of ammonia in the grab samples for the three urinals normalized by the total N concentration in the synthetic fresh urine recipe. Trap 1 and trap 2 samples are shown, with an additional data bar at the end for the concentration in the storage tanks at the end of the experiment. The results show that about 20% of urea was hydrolyzed in the mimicking experiment. The highest ammonia concentrations were seen in the storage takes at the end of the experiment, with trap 2 concentrations being higher than trap 1 concentrations throughout the experiment. This corresponds correctly with the assumption that urine was becoming more hydrolyzed because the progression of urine through the system allowed for more time for the urea hydrolysis reaction to occur. For

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the inhibition experiments, minimal concentrations of ammonia were measured, confirming that the urea hydrolysis reaction was inhibited by either decreasing the pH with acetic acid or increasing the pH with calcium hydroxide. The ammonia results are important because they provide unequivocal evidence that the CPS effectively monitored and controlled the extent of urea hydrolysis





Figure 3-2. Results for the mimicking and inhibiting experiments with no treatment, acetic acid addition, and calcium hydroxide addition. Normalized concentrations for ammonia, phosphate, calcium, magnesium, sodium, and potassium in grab samples taken every 30 min. Ammonia concentrations were normalized by the total N in synthetic urine. All other concentrations were normalized by the concentration in the synthetic urine at t = 0. Each bar is the average concentration from the three urinals or storage tanks. Trap 1 (orange bars), trap 2 (dashed bars), and the storage tank (white bars) are shown with error bars corresponding to one standard deviation.

The results for phosphate, calcium, magnesium, sodium, and potassium were used as a secondary check on urea hydrolysis progression. Fig. 3-2 shows the concentration of the ions at the sampling times normalized by the initial concentration in the synthetic fresh urine before the experiment began (C/C₀). As pH increases from pH 6 to 9 during hydrolysis, thermodynamically favorable conditions for precipitating struvite (NH₄MgPO₄·6H2O) and hydroxyapatite (Ca₅(PO₄)₃(OH)) are created (K. M. Udert, Larsen, & Gujer, 2003b). Therefore, losses in phosphate, calcium, and magnesium can be used to estimate the precipitative loss of nutrients within the system. The clear losses in calcium and magnesium in the mimicking urea hydrolysis experiment show the increased possibility of precipitation in the nonwater urinals. Additionally, a small loss of

potassium over time can be seen in Fig. 3-1. This could be due to the precipitation of the struvite potassium-analog (KMgPO₄·6H2O). Xu et al, 2015 found that the precipitation potential of the potassium-analog was higher than the precipitation potential of struvite at ammonium concentrations below 20 mM (Xu et al., 2015). In the inhibition experiments, a C/C_0 value of 1 means that there were no nutrient losses observed in the urine grab samples. It can be seen that the acetic acid and calcium hydroxide experiments were able to maintain C/C_0 closer to 1 than the mimic urea hydrolysis, despite the variation in the samples due to differences from urinal-to-urinal. The increase in calcium concentrations in the calcium hydroxide experiments was due to the addition of the inhibition solution. The sharp decrease in the phosphate at the end of the experiment is also due to the addition of calcium hydroxide, which forms a calcium phosphate precipitate as seen in the literature (Dyllon G. Randall et al., 2016). Using a chemical equilibrium software, it was confirmed that calcium phosphate, magnesium oxide, and magnesium phosphate minerals precipitate when using calcium hydroxide as a urea hydrolysis inhibition chemical (details found in SI).

The difference between the synthetic fresh urine and the storage tanks can be seen with more clarity in Fig. 3-S4. The figure shows the concentration of the measured ions for grab samples of the synthetic fresh urine at the beginning of the experiment, the storage tanks, and the synthetic fresh urine at the end of the experiment. The concentrations difference between the untreated synthetic fresh urine at the beginning and end of the experiment is minimal for the three experiments. As seen, the concentrations of ammonia are highest in the storage tanks for the mimicking urea hydrolysis experiment as expected. The phosphate results show that the concentrations in the storage tanks are the lowest when the urine was treated with calcium hydroxide, for the reasons mentioned above. Calcium concentrations are seen to be most stable when urine was treated with acetic acid, while there were losses when no treatment was used and elevated when calcium hydroxide was used. Magnesium followed a similar trend as calcium, with the exception that it was decreased in the storage tanks in the calcium hydroxide experiment. Sodium and potassium are seen to remain stable throughout the three experiments, because urea hydrolysis and subsequent precipitation does not affect the concentrations of sodium and potassium. Fig. 3-S4 concludes that acetic acid is a better chemical for urea hydrolysis inhibition in this context as it met the requirements to inhibit urea hydrolysis and to maximize the nutrient concentrations in the storage tanks. However, microbiology was not quantified during these experiments and future research should be conducted to determine the effects of acetate addition in urine diversion systems.



Figure 3-3. Mechanistic model results. Part a shows the results for the model using the parameters from the mimicking urea hydrolysis experiment. Ammonia and urea concentration results are shown for the modeled concentration inside the urinal trap (TU,
solid) and in trap 1 (T1, dashed). The experimental results for ammonia concentration from Fig. 3-2 are shown as triangles and squares. Part b shows the model results for variable urination volumes between 10–40 s. Part c shows the model results for variable urea concentrations between 400–600 mmol/L as N. Random selections in SI.

Ultimately, both base and acid addition were able to inhibit urea hydrolysis and they can both be used to inhibit urea hydrolysis in urine storage tanks (Flanagan & Randall, 2018; Hellstrom et al., 1999; Dyllon G. Randall et al., 2016). In some instances, such as stabilization of urea once urine enters the storage tanks, it could be beneficial to precipitate these minerals in situ using base addition, as they can be used as a form of nutrient recovery (Dyllon G. Randall et al., 2016; Simha, Senecal, et al., 2018). However, the long-term use of a calcium hydroxide slurry can lead to the adsorption of carbon dioxide by the slurry, creating calcium carbonate (Han, Yoo, Kim, & Wee, 2011). Alternatively, the addition of solid calcium hydroxide would require a redesign of nonwater urinals to allow for the fertilizer precipitate to be removed once it forms. Previous research applying solid calcium hydroxide has employed redesigned urinals that allow for the precipitate to be collected at each urinal, which would not fit the context of this study (Flanagan & Randall, 2018). As such, because the goal is to inhibit urea hydrolysis at the existing urinals and to decrease the possibility of precipitation throughout the collection pipes, it is important to consider the effects of the chemical addition on the functionality of the nonwater urinals and the pipes leading to the storage tanks. In the vision of urine diversion in this research on the scale of multi-story CI buildings with retrofitted drain pipe networks, it would be less difficult to implement the use of dilute acetic acid in the maintenance routine of nonwater urinals, than it would be to implement the addition of solid calcium hydroxide or a calcium hydroxide slurry. For

that reason, acetic acid was chosen as the urea hydrolysis inhibition chemical for the remainder of this study, with the acknowledgment that base addition could be a viable approach under suitable conditions.

Mechanistic and conceptual models of urea hydrolysis inhibition control

The model with rate constant fit from the experimental data was used to evaluate the effects of variable urination volumes and initial urea concentrations (Figure 3-3). The model showed that variability in ammonia concentrations was dampened as the urine moved from the urinal trap to trap 1 due to intermittent flow conditions, which resulted in a combination of plug flow and completely mixed behavior in the traps. The ammonia and urea concentrations has less variability in trap 1 than they were in the urinal trap for the conditions of variable urination volume and variable initial urea concentrations. The model shows that urea hydrolysis continues to advance in the three cases presented, showing that physiological variability, i.e. urination volume and urea concentration, cannot be used to control urea hydrolysis.

Fig. 3-4 shows the conceptual model for the effects of urination frequency and urine volume on urea hydrolysis progression. Fig. 3-4 illustrates that urea hydrolysis can occur by the combination of two factors. It begins in two settings: a low frequency restroom and a high frequency restroom (see dark boxes in Fig. 3-4). The frequency of use is a function of design and setting. The outcomes of these factors are seen in the white boxes. Urea hydrolysis tends to progress when low urine volumes and low frequencies of use are combined because the urination does not fully replace the urine from the previous urination and it has sufficient time for urea hydrolysis to progress. The urinal trap holds approximately 450 mL of liquid. If a urination is below that volume, it will mix with some of the urine held in the trap from the previous urination events. In contrast, the most of the urine in the trap will be replaced if the urination is greater than 450 mL. The combination of urine from the previous urination event and urine from the current urination creates a feedback loop for urea hydrolysis. Urea hydrolysis can also occur when low urination volumes are added at a high frequency due to the partial replacement of urine in the urinal trap. However, the amount of time between urinations can have an inhibitory effect on urea hydrolysis if urinations occur at high frequencies, no matter how low the urination volume.



Figure 3-4. Conceptual model for urea hydrolysis in nonwater urinals. The dark boxes indicate the two settings where urea hydrolysis can take place. The white boxes are the outcomes of urea hydrolysis under these two settings. The dark arrows indicate the reinforcing factors, i.e. low frequency and low volume or high frequency and high volume. The dashed arrows indicate balancing factors, i.e. low frequency and high volume or high frequency and low volume.

There are also two combinations of factors that lead to pseudo-inhibition of urea hydrolysis. It is called pseudo-inhibition because it does not use any urease enzyme inhibition mechanism that have been proposed in literature but urea hydrolysis does not progress as expected. Instead, it constrains the time allowed for the urea hydrolysis reaction to take place in the urinal trap. The first combination occurs when high volumes of urine are added at high frequencies, which replaces the entire volume in the urine trap at a high rate. Pseudo-inhibition can also occur when high volumes of urine are added at a low frequency because the urine in the trap is constantly replaced. Importantly, the CPS was created to test the conceptual model predictions presented in Fig. 3-4, which would be difficult to do without cyber components. Two control logics were used to inhibit urea hydrolysis using acetic acid addition. The urination volume and frequency where varied, within limits, to create conditions for urea hydrolysis progression and pseudo-inhibition. *Comparison of two control logics*

The next major objective was to use acetic acid as a urea hydrolysis inhibitor under more realistic conditions. Prior to these experiments, the experimental design was prescribed in that all urination events were the same volume, occurred at the same frequency, and inhibition chemicals were added after every urination event. Moving forward, the CPS allowed for varying of urination volume, urination frequency, and frequency of addition of the inhibition chemicals. The first set of experiments sought to compare two urea hydrolysis inhibition control logics: (1) a reactive control logic using a threshold for action and (2) a predictive control logic using predictive models for action. For these experiments, the pump controller was allowed to pick a urination frequency between 10–20 min, to mimic restrooms with low frequency of use. Because urine flow

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within the urinal testbed only occurs during urination events and the pH sensors are in the first trap, a 20 min lag was implemented after acid additions in order to avoid the addition of acid between the time of addition and the time it would reach trap 1. All experiments moving forward employed the same range for urination volume (10–40 s urinations). One limitation of this work was the inability to vary the flow rate, as it is expected that flow rate and urination volume will differ in the real world.



Figure 3-5. Results for the reactive control logic experiment using the low urination frequency experiment. pH in trap 1 for the three urinals is shown as circles. Urination events are shown by the green bars and the acid addition events are shown by the black bars. The height of the bars correspond to the urination time (between 10–40 s), which directly corresponds to the urination volume (118.5–450 mL). The acid addition bar height (20 s) corresponds to 2.5 mL acetic acid addition. The red dashed line corresponds to the pH threshold used for the reactive control (pH 7.0).

Fig. 3-5 shows the pH results for the reactive control logic experiment using the low urination frequency. The pH for all three urinals is displayed. The reactive control logic follows this rule: a message for acid addition was sent to the acid pump

immediately after 4 consecutive pH measurements (15 s per step) in trap 1 were above pH 7.0 for any of the three urinals. Fig. 3-5 shows that this occurred 6 times in approximately 6 hours. Therefore, the pH was controlled by the addition of acetic acid and it was not allowed to increase past pH 7 for an extended period of time. Two consecutive acid additions happened about 4 hours into the experiments, because the 20 min lag between acid additions matched the 20 min urination frequency that was randomly chosen for those two urination events. The acid addition from the previous urination was still in the urinal trap, and the pH sensor triggered an acid addition immediately after the 20 min lag was over. A urination within the 20 min lag would have pushed the acidified urine from the urinal trap into trap 1, decreasing the pH to below pH 7.0. Because that did not happen, the acid addition and urination event occurred at the same time.

Fig. 3-6 shows the pH results for the predictive control logic experiment using the low urination frequency. Acid addition was determined by the results of four predictive models made to predict the next 4 steps (15 s per step) in pH (Li, Saetta, Mirchandani, & Boyer, 2019). The models used 42 variables: the most recent 20 pH values, the most recent 20 conductivity values, the time value since the last urination event, and the time value for the length of the last urination event. Acid was delivered to the urinals immediately after all four models for any urinal predicted pH values above pH 7.0. As seen in Fig. 3-6, the acid was delivered 6 times during the 7 h experiment. The coincidence between the acid delivery lag and the longest time between urinations occurred two times, as seen in the cases where acid was delivered for two consecutive urinations. This is due to chance, as the program was written to randomly choose a length

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of time between urination events within the set range. The results from this experiment show that the pH increased past pH 7.0 more than it did in the reactive control logic experiment. The effects of that increased pH can be seen in Fig. 3-7, where the normalized concentrations of calcium and magnesium in trap 1 are shown. For the predictive control logic experiment, the concentrations of calcium and magnesium were lower than the concentrations in the reactive control logic experiment across the entire experiment because urea hydrolysis will allowed to proceed to a great extent, reaching pH as high at pH 8 and creating conditions for precipitation that were more favorable than they were at pH 7.



Figure 3-6. Results for the predictive control logic experiment using the low urination frequency experiment. pH in trap 1 for the three urinals is shown as circles. Urination events are shown by the green bars and the acid addition events are shown by the black bars. The height of the bars correspond to the urination time (between 10–40 s), which directly corresponds to the urination volume (118.5–450 mL). The acid addition bar height (20 s) corresponds to 2.5 mL acetic acid addition. The red dashed line corresponds to the pH threshold used for the predictive control (pH 7.0).



Figure 3-7. Normalized concentrations for phosphate, ammonia, calcium, and magnesium in grab samples from trap 1 taken every 30 min. Concentrations for phosphate, calcium, and magnesium were normalized by the concentration in the synthetic urine at t = 0. Concentration of ammonia was normalized by the total N concentration in the synthetic urine recipe. Each bar is the average concentration from the three urinals. Reactive control logic experiment (blue bars) and predictive control logic experiment (dashed bars) are shown with error bars corresponding to one standard deviation.

concentrations of ammonia, phosphate, calcium, magnesium, sodium, and potassium for grab samples taken from trap 1 and trap 2 every 30 min throughout the experiment, with an additional sample of the storage tanks at the end of the experiment. Based on the ammonia concentrations, it can be seen that both the reactive and the predictive control logic experiments with low urination frequencies were able to inhibit urea hydrolysis. However, the predictive experiment allowed the pH to increase to as much as pH 8,

In terms of urea hydrolysis inhibition, Fig. 3-S7 shows the normalized

which created conditions for more hydroxyapatite and struvite to precipitate with the 15 mmol/L of NH₃ that had formed from the small amount of urea hydrolysis that occurred. The reactive control logic experiment was able to keep the concentrations of ions more stable, which could be attributed to the pH staying more stable throughout the experiment.

Comparison of two restroom use frequencies

The last objective of this study was to compare the use of urea hydrolysis inhibition control with two restroom use frequencies. The low frequency reactive control logic experiment that was discussed above was compared with the high frequency reactive control logic experiment (see Fig. 3-8). The pump code was changed to allow for urinations every 1-10 min, compared with the 10-20 min frequencies used in the previous experiments. Figure 3-7 shows that acid was only delivered one time during the 5 h experiment. The high urination frequency did not allow sufficient time for urea hydrolysis to occur, keeping the pH below 7.0 for the majority of the experiment. The pH was also affected by the volume of the urinations, as it is evident that high volume urinations in high frequencies decreased the pH. The opposite case, low volumes with the lowest frequencies allowable, increased the pH to above 7.0, triggering the acid addition. The volume and frequency of addition were contributing to a change in the flow patterns in the testbed. High volumes at high frequencies were acting as a plug flow throughout the system. Low volumes at low frequencies were able to create a system with characteristics of a continuously mixed flow reactor. The effect of the high use frequency can be seen in Fig. 3-S8. The cumulative volume shows that close to double the volume was introduced into the urinals in less time than the low use frequency experiments. The

time needed for urea hydrolysis was not attained; therefore most of the urine went through the testbed unchanged from its initial composition. Fig. 3-S7 shows that urea hydrolysis did not occur but acetic acid was not added for most of the experiment. A pseudo-inhibition occurred due to the manipulation of time between urination events.



Figure 3-8. Results for the reactive control logic experiment using the high urination frequency experiment. pH in trap 1 for the three urinals is shown as circles. Urination events are shown by the green bars and the acid addition events are shown by the black bars. The height of the bars correspond to the urination time (between 10–40 s), which directly corresponds to the urination volume (118.5–450 mL). The acid addition bar height (20 s) corresponds to 2.5 mL acetic acid addition. The red dashed line corresponds to the pH threshold used for the predictive control (pH 7.0).

Implications

The results of this research show that cyber-enabled nonwater urinals, and eventually UDTs, can be monitored and controlled to inhibit urea hydrolysis, which has direct benefits for water conservation in traditional plumbing and would benefit nutrient recovery in urine diversion mode. As a CPS, the acid addition can be automated to

respond to changes in pH and other important variables in the urinal system. Depending on the use of the restrooms, acid would only be delivered to the urinals about once per hour, and because this would be fully automated, no manual labor (e.g., custodial staff) would be required. For restrooms with high use such as high traffic common areas, acid would only be added during times of low use, such as nighttime or weekends, further reducing the amount of acid needed for the urinals. For nighttime and weekends, the CPS could be programmed to deliver the amount of acid needed to maintain the pH of the urine inside the trap below pH 4. A redesign of the urinal to include pH sensors inside the trap would increase the capabilities of the CPS by providing a monitoring point closest to the fresh urine. If the urinal is not redesigned, the CPS could be instructed to "sleep" during the nighttime, with the informed assumption that the urine in the urinal trap is acidified but not reaching the pH sensor outside of the urinal. Both methods of nighttime urea hydrolysis control would be difficult to implement if a CPS is not employed.

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CHAPTER 4

IMPACT OF ACETIC ACID ADDITION ON NITROGEN SPECIATION AND BACTERIAL COMMUNITIES DURING URINE COLLECTION AND STORAGE

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Abstract

The rate of urea hydrolysis in nonwater urinals is influenced by the volume of urination events and the frequency of urinal use. Inhibition of urea hydrolysis with acetic acid addition has been demonstrated at the laboratory scale but it was not able to fully represent the conditions of a real restroom with real urine collection. The goal of this study was to understand the effects of acid addition for control of urea hydrolysis on nutrient concentrations and bacterial communities in human urine during collection and storage. Three control logics were used to determine the schedule of acid addition: (i) acid addition after every urination event, (ii) acid addition during periods of high building occupancy, and (iii) acid addition during periods of low building occupancy. Wifi logins were used to approximate building occupancy and to create the control logics used in the study. All three control logics were able to inhibit urea hydrolysis. The bacterial communities were identified to determine the impact of acid addition on the community structure. The collection of urine by nonwater urinals alone did not reduce the presence of enteric bacteria commonly found when collecting urine with urine-diverting toilets. Acid addition reduced the community diversity and created conditions for higher relative

abundances of the order Enterobacteriales. Finally, results from stored acidified urine showed that urea hydrolysis inhibition is reversible and is influenced by the amount of acid added at the urinal. The amount of acid added can influence the rate of hydrolysis in the storage tanks and can be used to select for urea- or ammonia-nitrogen for nutrient recovery. This study is the first of its kind to inhibit urea hydrolysis in nonwater urinals in a real restroom with real urine, and is the first to identify the bacterial communities in urine collected solely with nonwater urinals.

Introduction

Urine diversion has the possibility of dramatically altering wastewater collections systems in commercial and institutional (CI) buildings, led by its benefits towards water conservation and resource recovery (Larsen & Gujer, 1996). Urine diversion has been proposed as a sustainable source for nutrients and fertilizers, for the ability to target pharmaceuticals, and for its connections to water conservations (Boyer & Saetta, 2019; Patel et al., 2020; D. G. Randall & Naidoo, 2018). Life cycle assessments have also shown that urine diversion can be equally or better than conventional wastewater treatment in terms of environmental impact (S. K. Ishii & Boyer, 2015; Landry & Boyer, 2016). It is now imperative to move towards the transition from laboratory-scale development to full-scale operation of urine diversion systems. The vision for urine diversion systems in CI buildings is as follows. Urea hydrolysis inhibition is needed at the point of collection, regardless of downstream treatment in order to remove the collection fixtures as points of failure in the systems (Boyer & Saetta, 2019; D. G. Randall & Naidoo, 2018). For example, in the United States, plumbing codes have been amended to include nonwater urinals (American Society of Mechanical Engineers, 2006), providing the opportunity for implementation of urine diversion without needing additional codes for the collection fixtures. Next, based on stakeholder needs, on-site storage and treatment would produce location-specific products that can enter the local supply chain. Sensors and automation throughout the collection and treatment system would reduce the amount of labor needed on-site, such as cyber-physical systems (CPSs) proposed for urea hydrolysis inhibition (Saetta et al., 2019b). This vision allows for less change to the conventional wastewater system within buildings and thus reducing the

need for altering behaviors by building occupants. This vision relies on only the collection of urine from nonwater urinal users in order to comply with the US plumbing codes. Using nonwater urinals is the prime opportunity to implement urine diversion while the plumbing codes are revised to allow for urine-diverting toilets across the US, at which point the vision would include collection from all users.

Hence, functionality of collection fixtures is necessary in order to fulfill this vision of urine diversion. Improving the function of nonwater urinals and developing urine-diverting toilet technology has been identified as a research need in the urine diversion literature (Boyer & Saetta, 2019; Larsen, 2020; McConville, Kvarnström, Jönsson, Kärrman, & Johansson, 2017). Malfunction occurs as the urea hydrolysis reaction creates conditions for precipitation of urine salts in the fixture. Acid addition at the urinal has been shown to inhibit urea hydrolysis in synthetic urine (Hannah Ray et al., 2017; Saetta & Boyer, 2017; Saetta et al., 2019a, 2019b). Urea hydrolysis is the enzymatic reaction which hydrolyzes urea into ammonia and bicarbonate, raising the pH from 6 to 9 (K. M. Udert, Larsen, Biebow, et al., 2003). Acid addition lowers the urine pH to below the optimal pH for the urease enzyme (Krajewska, 2009). Additionally, the lowered pH reduces the precipitation potential of struvite (NH4MgPO4·6H2O) and hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂) by shifting the speciation of PO₄³⁻ to H₂PO₄⁻. Research has also shown the use of base addition to inhibit urea hydrolysis by increasing the pH past the optimal pH (Dyllon G. Randall et al., 2016; Senecal & Vinnerås, 2017; Simha, Senecal, et al., 2018). This technique is more amendable to a urine diversion system where nutrient recovery occurs at the urinal or toilet due to the co-benefits occurring insitu, such as the precipitation of calcium phosphate (Flanagan & Randall, 2018). As such, acid addition reduces precipitation that would otherwise hinder the functionality of existing nonwater urinals that are targeted in this research.

In Saetta et al. 2019, a conceptual model for urea hydrolysis identified two major factors that lead to urea hydrolysis progression: (1) frequency of urinal use and (2) volume of urination events (Saetta et al., 2019a). A urinal in a restroom with high use frequencies, e.g. in an airport, may need minimal acid addition due to the fact that fresh urine is readily replenishing the urine in the urinal trap. Inversely, a urinal in a restroom with low use frequencies, e.g. in a seldom occupied corner of an office building, may require more frequent acid addition, as urine in the trap is not readily replaced with fresh urine. While the volume of each urination is difficult to measure, the use frequency can be measured by motion sensor or other occupancy detection technology. This paper proposes the use of motion sensors to directly measure the use frequency. However, it is understood that motion sensors cannot be ubiquitously implemented to all nonwater urinal systems due to a vast landscape of urinals with different designs and CI buildings that cannot accommodate for additional technical expertise. Therefore, Wifi technology is proposed as a predictor for urinal use frequency.

The proposal for using Wifi logins to predict occupancy has been used by energy CPSs in buildings to increase energy savings (W. Wang, Hong, Li, Wang, & Chen, 2019), as Wifi logins have been shown to correlate with energy load increases (J. Chen & Ahn, 2014). Recently, Wifi logins have also been applied to drinking water systems within CI buildings, as its tied to water use and water quality (Richard, Hamilton, Westerhoff, & Boyer, 2020). Wifi logins may also be connected to urinal use and could be used as a manner for approximating the urinal use by way of building occupancy. Inherently, multiple benefits exist for using Wifi as a predictor of occupancy, including it being a low-cost data set, it being widely available, and it being a standard method for communication within buildings. Hence, a system using Wifi logins as a predictor of occupancy can be easily transferred to multiple buildings with the same algorithms.

A CPS for urea hydrolysis inhibition has the ability to control urine chemistry (Saetta et al., 2019b). However, the impacts on the microbiology present in urine has not been studied, especially as they pertain to the addition of acetic acid. Contrary to general assumptions, new microbiology tools have shown that urine is not sterile (Hilt et al., 2014; Wolfe & Brubaker, 2015). Studies have shown that urine from healthy men and women contain a microbiota that is unique to the bladder and urethra (Gottschick et al., 2017). Immuno-compromised individuals may also excrete pathogens, such as those related to urinary tract infections (Schönning, 2001). Additionally, urine may become cross-contaminated by fecal material during the collection process in traditional urine diversion systems (i.e., where at least a portion of the urine is collected using toilets). This is the first study of its kind to collect urine solely from nonwater urinals in a public restroom. In theory, this will reduce the amount of fecal contamination. However, it is unknown whether the restroom ecosystem and toilet plumes will have an impact on the urine bacterial community (Johnson, Lynch, Marshall, Mead, & Hirst, 2013). Höglund et al. 1998 found that some bacteria, such as *E. coli* died off rapidly during hydrolysis, while others, such as fecal *streptococci*, persisted during storage (Hoglund, Stenstrom, Jonsson, & Sundin, 1998). Hence, there is an interest to understand the changes in bacterial community structure after acid addition to inform the community about the benefits or risks of acid addition on the presence of harmful or pathogenic bacteria.

This research proposes the use of acetic acid to inhibit urea hydrolysis, which inherently inhibits the formation of biocidal ammonia (Hellstrom et al., 1999; Hannah Ray et al., 2017; Saetta & Boyer, 2017; Saetta et al., 2019a). Additionally, the addition of the simple organic carbon in acetic acid can alter the bacterial life in urine storage tanks. Acetic acid has a pK_a of 4.75. At pH below the pK_a, acetic acid is able to enter to bacterial cells by diffusion, where the internal pH is above the pK_a. Once inside the cell, the acid deprotonates, pH inside the cell decreases, and acetate accumulates until cell death occurs (Trček, Mira, & Jarboe, 2015). However, at pH above the pK_a, acetic acid deprotonates in the bulk liquid and acetate acts as a carbon source for microbial life. For example, acetic acid is used in wastewater treatment as an external carbon source for nitrogen removal, which stimulates the growth of biomass in the system (US Environmental Protection Agency, 2013). Therefore, monitoring the pH of urine storage tanks receiving acetic acid is necessary to understand the relationship between the pH and pK_a of acetic acid in terms of bacterial inhibition and growth.

With all of this in mind, this paper serves to inform the future implementation of urine diversion systems in CI buildings by taking the next step towards radical change of our wastewater treatment landscape (Kemp, Schot, & Hoogma, 1998). As such, the goal of this research was to understand the effects of acid addition for control of urea hydrolysis on nutrient concentrations and bacterial communities in human urine during collection and storage. The specific objectives were to quantify the impacts of acetic acid addition on (1) urine chemistry during collection, (2) bacterial communities during collection, and (3) urine storage. In this research, collection is defined as dynamic process where fresh urine is added to the system whereas storage is defined as static process where no fresh urine enters the system. Experiments were conducted using a novel "urinal-on-wheels" that was wheeled daily into a public restroom to monitor and control urine conditions in real-time during collection (See Figure 4-S1 and 4-S2). Following collection, the urine was stored for 120 days to monitor further changes in composition.

Materials and Methods

Urinal-on-wheels

A Sloan Water-Free urinal and 17 gal (64 L) storage tank was mounted on a metal frame with wheels to create the "urinal-on-wheels" (see pictures in Supporting Information (Appendix B), Fig. 4-S1 and 4-S2). Human urine collection was approved by the Arizona State University Institutional Review Board. The urinal was hung on the outside of the box, while the tank, sensors, microcontroller, and battery were housed inside the box. The collection tank was outfitted with Atlas Scientific pH and temperature sensors for real-time monitoring. An OSEPP Passive Infrared Sensor (PIR) Sensor and an URTONE UR198 Momentary Push Button Switch was placed on the outside wall above the urinal to count the number of users (users were asked to press the button after donating urine). An Atlas Scientific EZO-PMP peristaltic pump was used for acetic acid delivery into the urinal for acid addition experiments. The sensors and pump were controlled using a Raspberry Pi 3 Model B with a python-coded program. Data was stored locally on the microcontroller SD card. Measurements for the four sensors were logged every 5 min. The urinal-on-wheels was placed inside a multi-stall restroom in a seven-story institutional building on the Arizona State University campus. The restroom had 2 water-flushing urinals and three toilets. A "run" consisted of 2 weeks (Wednesday to Wednesday) of urine collection, with the urinal in the restroom for at least 4 h per day.

The urinal was not placed in the restroom during the two weekends (Saturday and Sunday) in the run period but the sensors were used to monitor the urine in the tank during the weekends. The urinal-on-wheels was stored in a laboratory space during nighttime and weekends. With this experimental design, runs consisted of 11 days of collection, in which collection was a dynamic process. The Wifi login data was collected by the Arizona State University Information Technology (ASUIT) and was reported as the sum of all Wifi logins per hour that occurred on the restrooms' floor level.

Four experiments were conducted: (1) monitoring urea hydrolysis, (2) acetic acid addition after every urination, (3) acetic acid addition when occupancy is above the occupancy threshold, and (4) acetic acid addition when occupancy is below the occupancy threshold (see Table 4-1). Each experiment had two runs. The first experiment monitored urea hydrolysis, meaning that no acetic acid addition was delivered to the urinal and the urea hydrolysis reaction was allowed to progress. The second experiment delivered acid to the urinal after each time the button was pushed. The third and fourth experiments added acid based on the occupancy threshold selected. The urinal-on-wheels was not inoculated with anything prior to experiment 1. Between runs, the urine tank was emptied but it was not cleaned. The tank used for Runs 1 and 2 (no acid addition) was replaced with a new tank for Runs 3 and it was used for Runs 3–8 (acid addition). The Sloan urinal has a removable cartridge (Falcon Water Technologies Velocity Cartridge) that acts as the p-trap. A new cartridge was used for each run and fresh sealant was poured into the trap at the beginning of each run.

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	Experiment	Experiment	Experiment 2	Experiment	Experiment	Experiment 4
	1	1b		2b	3	
Name	Urinal-on-	Glass Urinal:	Urinal-on-	Glass	Urinal-on-	Urinal-on-
	wheels: Urea	Urea	wheels: Urea	Urinal: Urea	wheels: Urea	wheels: Urea
	hydrolysis	hydrolysis	hydrolysis	hydrolysis	hydrolysis	hydrolysis
			inhibition	inhibition	above	below
			acetic acid		threshold	threshold
Total Length	2 weeks	20 days	2 weeks	20 days	2 weeks	2 weeks
Name of Runs	Run 1 & 2	_	Run 3 & 4	_	Run 5 & 6	Run 7 & 8
Number of	0	0	1	0	1	1
Pumps						
Pump details			Pump = acetic		Pump =	Pump = acetic
(flow rate)			acid (7.5		acetic acid	acid (7.5
			mL/min)		(7.5 mL/min)	mL/min)
Urination events	Random use	2 urination per	Random use	2 urination	Random use	Random use
timing		day		per day		
		Urination = 37	Acetic acid	Urination =		
Constant inputs		mL	soln, 2.5 mL	37 mL		
(frequency)			(every time	Acetic acid		
			button is	= 0.39 mL		
			pressed)			
Variable inputs					Acetic acid	Acetic acid
(frequency)					soln, 2.5 mL	soln, 2.5 mL
					(every 1 h	(every 1 h
					during	during periods
					periods of	of low
					high	occupancy)
					occupancy)	

Table 4-1. Experimental details

Urine samples for nutrient concentration analysis were taken at the end of each collection day within 15 min when the urinal is taken out of the restroom. The tank mixed on the way back from the restroom due to the movement from the restroom to an elevator and to the laboratory space for storage. Samples were taken inside the laboratory space to comply with biohazard rules. The samples were acidified with sulfuric acid to pH < 2 and were stored at 4°C until analysis of the samples was conducted. Prior to analysis, samples were filtered using 0.2 μ m nylon syringe filters (Environmental Express). Samples were diluted by 100× for phosphate and ammonia analysis with a Lachat Flow Injection Analyzer and by 1000× for total nitrogen (TN) and total organic carbon (TOC) analysis using a Shimadzu TOC-TN analyzer.

At the end of the collection period, urine samples for microbiology analysis were taken for Runs 1–8. Four 50 mL samples were collected into Falcon tubes. Samples were centrifuged at 4000 G for 12 min. The pellet was removed and placed into a 1.5 mL centrifuge tube. Pellets were stored at –20°C for short-term shortage and –80°C for long-term storage until all samples from the runs were collected. To study the storage period, a 2L urine sample was taken at the end of each acid addition run (Runs 3–8) to assess changes to urine chemistry for urine storage conditions. The samples were stored at room temperature in 1 gal (3.8 L) clear glass bottles. pH and conductivity measurements were taken once per week during storage. Periodic grab samples were taken from the storage bottles for TN, TOC, ammonia, and phosphate measurements. The methods above for analysis were followed for the storage samples. In terms of experimental timelines, day 0 of storage began on day 11 for Runs 3–8. The storage samples were stored and sampled for 120 days.

Glass urinal

A glass urinal was made by the Arizona State University Glassblowing Facility (see picture in Supporting Information (Appendix B), Fig. 4-S3). It consisted of a 2L round bottom flask (or "tank") with two joints. One joint was used as the inlet from the urinal trap to the flask. The second joint was used as the vent to allow for air to pass and for urine to flow through the glass urinal. The glass urinal trap was blown to hold approximately 70 mL of fluids and it has a funnel shaped top to catch the urine as it is poured into the trap. Based on the dimensions of the trap, the corresponding fluid volumes (urinal sealing liquid and urine held in the trap) were calculated to match the ratios of the fluids in the real nonwater urinals used in Saetta and Boyer (Saetta et al., 2019a). Kohler Waterless Urinal Sealing Liquid (containing >80% vegetable oil, > 10% polyamide gel, and >10% proprietary blend of nonhazardous components) was used as the sealing liquid (Kohler, 2015). The ratios used in the glass urinal were 14 mL of sealing liquid and 37 mL of urine per urination event.

Two experiments were conducted with the glass urinal (see Table 4-1). The first experiment was conducted to monitor urine chemistry and microbiology in urea hydrolysis conditions. The second experiment was conducted to monitor the urine chemistry and microbiology in acetic acid addition conditions. Each experiment was conducted three times to assess accuracy. The runs consisted of 3 weeks of urine collection. Urine donors were asked to collect urine in sterilized urine collection cups in a nearby restroom. Urine was added as soon as collected to ensure freshness. Urine was added to the glass urinal twice per day. The pH and conductivity of the fresh urine samples was taken prior to the urine being poured into the urinal (see Figure 4-S8). During each run, a 30 mL sample of the urine in the collection flask was taken at the beginning of every Monday and at the end of every Friday. The pH and conductivity of the flask samples was taken immediately after collection.

At the end of each run, a 20 mL sample was taken from the flask. The sample was acidified with sulfuric acid to pH < 2 and was stored at 4°C until further TN, TOC, ammonia, and phosphate analysis using the methods described in the section 2.1 (see Figure 4-S9). The flask was magnetically stirred at 400 rpm for 3 min to allow for homogenization of the urine solution and a 50 mL Falcon tube was filled for pellet recovery. The samples was centrifuged at 4000 G for 12 min and a pellet was recovered and stored at -20° C for short-term storage and -80° C for long-term storage until all experiments are finalized.

DNA extraction and data analysis

DNeasy Powersoil Kit were used for DNA extraction of the frozen pellets and instructions were followed. A BioTek Synergy H1 Hybrid Multi-Mode Reader was used as a quality check to determine the concentration of DNA extracted in the samples. DNA sequencing was conducted by the ASU Biodesign Institute Genomics Facility. Bacterial community analysis was performed via next generation sequencing in MiSeq Illumina platform. Amplicon sequencing of the V4 region of the 16S rRNA gene was performed with the barcoded primer set 515f/806r designed by Caporaso et al. 2011 and following the protocol by the Earth Microbiome Project (EMP) for the library preparation (Caporaso et al., 2011). PCR amplifications for each sample were done in triplicate, then pooled and quantified using Quant-iTTM PicoGreen dsDNA Assay Kit (Invitrogen). A no template control sample was included during the library preparation as a control for extraneous nucleic acid contamination. 200 ng of DNA per sample were pooled and then cleaned using QIA quick PCR purification kit (QIAGEN). The pool was quantified by Illumina library Quantification Kit ABI Prism[®] (Kapa Biosystems). Then, the DNA pool was diluted to a final concentration of 4 nM then denatured and diluted to a final concentration of 4 pM with a 25% of PhiX. Finally, the DNA library was loaded in the MiSeq Illumina and run using the version 2 module, 2x250 paired-end, following the directions of the manufacturer. Data was analyzed using QIIME2 (Quantitative Insights Into Microbial Ecology), which was used to determine the relative abundance of bacterial communities in the samples (Bolyen et al., 2018).

Results and Discussion

Urine chemistry during collection

Wifi logins were used as a surrogate measurement of building occupancy for the urinalon-wheels experiments. Saetta et al. concluded that urea hydrolysis could be controlled based on frequency of urinal use (Saetta et al., 2019a). Using building occupancy to create control logics would eliminate the use of sensors at the urinal, simplifying acid addition at the urinal for urea hydrolysis inhibition and eliminating the cost and maintenance of the sensors. However, most buildings lack occupancy sensors. Wifi has been used as a surrogate for occupancy in energy CPSs (W. Wang et al., 2019) and water quality studies (Saetta, Richard, Leyva, Westerhoff, & Boyer, 2020) in buildings, as it provides an accurate estimation for building occupancy. In this case, control logics were created using historic data from the year before the experiments were conducted. Fig. 4-1 shows the historic Wifi logins per day for the specific floor where the experiments were conducted (September 2018 to August 2019). Weekly patterns are easily seen in these data, as well a dramatic decrease in logins during the December holiday break and a decrease in the summer months. The 70th, 80th, and 90th percentile of the data was taken and the resulting values for the total data set, fall, spring, and summer semesters are seen in the table insert in Fig. 4-1. The Wifi login values would indicate the point at which the building went from low to high occupancy based on the percentiles calculated. Ultimately, the 80th percentile values for the specific semesters were chosen as the cut-off value for the urinal-on-wheels experiments. The average number of Wifi logins per hour per day can be seen in Fig. 4-S4.



Figure 4-1. Time series of the floor-specific Wifi logins per day for September 2018 to August 2019. Wifi login thresholds are displaying on the graph for the total time series, the fall, spring, and summer semesters. The 80th percentile values were chosen for this study.

The proposed Wifi logins method could be implemented in a similar manner with real-time data. Daily averages can be calculated by algorithms at the edge, meaning that the algorithms are located as close to the sensors as possible (Corcoran, Lemley, Costache, & Varkarakis, 2019), which would be compared to real-time Wifi login data to determine the acid addition schedule with as much specificity as desired, e.g. logins per building by aggregating logins for all Wifi access points in the building, logins per floor by aggregating logins for all Wifi access points on each floor, or logins per restroom by selecting Wifi access points nearest to each restroom. The specificity to which the control logics are designed would depend on the use frequency of each restroom and the difference from restroom to restroom. This research assumes that the number Wifi logins influences frequency of urinal use, when in reality, a Wifi user can enter a building a never use a urinal. However, the trends taken calculated from the Wifi logins do not have to be granular in their measurements. The Wifi logins are used as a measure of periods of high and low occupancy. The assumption lies on the idea that more people will use the urinals during high occupancy than during low occupancy and that is why the Wifi logins provide a good measurement without needing additional intervention around each urinal.

Four urinal-on-wheels experiments were conducted (2 runs per experiments for a total of 8 runs). The objective of the first experiment was to monitor the extent of urea hydrolysis occurring in the urinal-on-wheels without acid addition. These runs served as a check on the monitoring equipment and the design of the urinal-on-wheels. Three subsequent experiments were conducted to test three acid addition control logics: (1) Run 3–4 tested acid addition after every urination event by asking donors to press a button after using the urinal, (2) Run 5–6 tested acid addition once an hour during periods of high floor occupancy, and (3) Run 7–8 tested acid addition once an hour during periods of low floor occupancy. During Runs 5 and 6 (acid addition during periods of high occupancy), acid was added between 4 and 9 times per day because the experiments were conducted in the summer semester and historically, the number of Wifi logins were above

13 during 4 to 9 hours of the day. For Runs 7 and 8 (acid addition during periods of low occupancy), acid was added between 15 to 24 times per day because those experiments were conducted in the fall semester and historically, the number of Wifi logins were below 28 during 15 to 24 hours of the day.

First, the urinal-on-wheels was able to provide interesting results when it comes to use patterns and the efficiency of the acid addition logics. Fig. 4-2 compares the outcomes of the all 8 runs. Part (a), (b), and (c) shows the use patterns and average urination volume for each run. Parts (a) and (b) show that the number of users varied widely between runs, even though the urinal-on-wheels was placed in the restroom for an equal amount of time across all runs. The average urination volume for all the runs was 283.3 ± 38.6 mL, which is within 50 mL of the average urination volume of 237 mL that has been reported in the literature (Latini, Mueller, Lux, Fitzgerald, & Kreder, 2004). Interestingly, the urination volumes were the lowest when the ambient air temperatures of Arizona were the highest in mid-Summer, confirming that urination volumes are negatively correlated to air temperatures because increased temperatures increase water loss due to sweat but does not increase water intake (Mora-Rodriguez et al., 2016). And although not the focus of this research, the ability to monitor urination volume and composition suggests the potential to monitor human health conditions such as hydration and dehydration.



Figure 4-2. Comparison of all treatments. (a) Total number of users per run as indicated by the button push. (b) Total time the urinal-on-wheels was in the restroom per run. (c) Calculated urination volume per urination event based on total volume collected and number of users per run. (d) Total volume of acid added per run. (e) Calculated volume of acid added per urination event per run. (f) Calculated percent of acid added to the urine tank by volume. (g) Final pH measurement of the urine tank per run. (h) Final ammonia concentration in urine grab samples taken at the end of Day 11. (i) Final phosphate concentration in urine grab samples taken at the end of Day 11. Error bars for duplicate analysis are shown with the standard deviation for (h) and (i).

Parts (d), (e), and (f) show the results for acid addition. As expected, the least amount of acid added to the urinal was seen during experiments testing addition at periods of high occupancy. This is because acid was only added once per hour for 6 to 9 hours per work day depending on the day and there was no addition during the weekends. The opposite effect can be seen with the acid addition results for Runs 7 and 8. Acid was added once per hour at periods of low occupancy, which includes nights and weekends. Part (e) was used as a method of calibrating the acid addition. Whenever the pump was activated, whether due to button push or occupancy logics, 2.5 mL of acetic acid was added. The actual total addition of acid was impacted by interruptions in operation, such as loss of power or program failure. Ideally, the volume of acid per user for Runs 3 and 4 would be 2.5 mL, as that was what the program was coded to deliver. As calculated, 2.4 mL and 2.3 mL of acid were added in Runs 3 and 4, respectively. This is less than 10% different than the desired 2.5 mL of acid, therefore the method of acid addition was determined to be adequate for future runs. The greatest amount of acid added per urination event was seen in Runs 7 and 8 at 5.0 mL and 5.5 mL, respectively. That is more than 2 times the amount of acid added during runs of acid addition after every urination event. The implications of these results will be discussed subsequently.

Parts (g), (h), and (i) show a snapshot of the results in terms of urea hydrolysis inhibition. The pH at the end of each run is seen in part (g). As expected, the pH during urea hydrolysis monitoring experiments was 8.85 and 8.93 for Runs 1 and 2, respectively. During acid addition experiments (Runs 3–8), the final pH was between 4.56–5.54. This pH range spans the pK_a of acetic acid of 4.75. Part (h) shows the concentration of ammonia in the Day 11 sample taken from the urinal tank (expanded data for seen in Fig. 4-3). As a result of acid addition, the average concentration of the final ammonia samples for Runs 3–8 was 20.2 ± 2.2 mmol/L as N, meaning that urea hydrolysis was inhibited by all three acid addition logics. This concentration falls within the range of ammonia in fresh urine given in the literature: 12–42 mmol/L as N (Putnam, 1971). Part (i) shows the concentration of phosphate in the Day 11 sample taken from the urinal tank. The acid addition did not affect the phosphate concentration, as expected. The increase in pH in Runs 1 and 2 should have led to the precipitation of phosphate as struvite (NH₄MgPO₄· 6H₂O) but that was not evident in the final concentration of phosphate in the Day 11 samples (K. M. Udert, Larsen, & Gujer, 2003b). There was no observed precipitate in the samples but there was precipitate on the sensors when they were cleaned after between runs. The trends in the measured phosphate concentrations could be due to the sampling method, mainly because acidifying the sample upon collection could have dissolved any precipitated phosphate. Overall, the acid addition was able to inhibit urea hydrolysis in real urine as seen in the urine chemistry results. This confirms the trends seen in synthetic urine urea hydrolysis experiments (Saetta & Boyer, 2017; Saetta et al., 2019a).

Fig. 4-3 shows the pH of urine during each run. For Runs 1 and 2, the pH increased rapidly from pH 7 to pH > 8.5 within the first week in use, where it is then buffered at pH 9. The increase in pH is indicative that urea hydrolysis was happening, but it does not give an indication of the extent of hydrolysis due to the buffering at pH 9 (Hannah Ray et al., 2017). The pH in the acid addition experiments (Runs 3–8) can be seen to react to the logics in different ways. Because the sensors were in the urine tank, the effects of acid addition on pH in the tank was dependent on the frequency of urinal use. In Runs 3 and 4, acid was added after every urination event, leading to a constant pH throughout runs because acid was constantly added and entering the tank with the incoming urinations.



Figure 4-3. Urine pH measurements taken every 5 min for Runs 1–8. In the legend, D denotes days, N denotes nights, and W denotes weekends.

In Runs 5 and 6, acid was added at periods of high occupancy, leading to less consistency in the pH when compared to the the pH in Runs 3 and 4. However, the acid was able to enter the tank during periods when the urinal was being used. In Runs 7 and 8, acid was added at periods of low occupancy, leading to a build up to acid in the urinal trap. This build up would only enter the tank once the urinal was used during the workdays and the acid would then build up again at night. The pH responded to this acid addition logic with the most inconsistency. Conductivity was not used in the study because the probe malfunctions with small amounts of scaling across the sensing window.

The temperature sensor in the tank monitored the conditions in the tank and the results show an interesting parameter that could be used to monitor urine storage tanks in future applications. The temperature in the storage tank reacted to the daily use of the urinal and the volume of urine collected (see Fig. 4-S5). Urine is excreted at body temperature (37°C). As more urine is collected, the temperature inside the storage tank rises. At night, when urine is not collected, the stored urine temperature decreases to room temperature (22°C). As the volume of urine in the tank increases, the influence of fresh urine on the tank temperature decreases. However, changes in temperature were still evident on the last day of the runs. Hence, variance in temperature of urine in small to medium collection tanks could be used in conjunction with occupancy sensing (e.g., Wifi) to control urea hydrolysis via acid addition.

Fig. 4-4 shows the concentration of ammonia, TN, and the calculated concentration of urea for daily urine samples in each run. The concentrations for samples during Runs 1 and 2 confirm that urea hydrolysis was occurring, as the ammonia concentrations increase from day 1 to day 11. The coefficient of variation for ammonia concentration in these runs was 44% and 39%, respectively. Comparatively, the coefficient of variations for ammonia concentration in the acid addition runs (Runs 3–8) were below 15%, with the lowest coefficient found in Run 8 at 4%. These results are direct measurements of the urea hydrolysis reaction and are better indicators of the extent of urea hydrolysis (Hannah Ray et al., 2017). The percent of urea hydrolysis based on the reported measurements was between 4.6–6.4% for Runs 3–8. Run 3–8 were all able to

inhibit urea hydrolysis, while Runs 1 and 2 showed increasing extents of urea hydrolysis. Fig. 4-S6 and 4-S7 show the results for phosphate and TOC in a similar fashion.



📕 Ammonia 📕 TN 📘 Urea calc

Figure 4-4. Ammonia and total nitrogen (TN) measurements of daily urine samples during urine collection for Runs 1–8. Urea is calculated as the difference between TN and ammonia concentrations.

This study was designed to inform future implementation of urine diversion systems in CI buildings. The acid addition allows for control of nitrogen speciation, specifically allowing for a decision between inhibiting urea hydrolysis and allowing hydrolysis to ammonia in the stored urine. The amount of acid added had an impact on the concentrations of urea and ammonia in the stored urine and the time to hydrolysis during storage. It is conceivable to design a system which inhibits urea hydrolysis at the urinal and storage tank to enable urea recovery (Hannah Ray et al., 2019) or inhibit urea hydrolysis at the urinal but allow for complete hydrolysis at the storage tank to enable ammonia recovery (H. Ray, Perreault, & Boyer, 2020). This allows for tailoring suite of technologies that could be used to recover products based on local needs. With this in mind, it is also important to account for the amount of acid needed based on economic impact for the raw materials and the labor needed to replenish acid storages at the urinals. However, when the recovery of urea is more economically favorable in a specific location or situation, the extra addition of acetic acid to keep hydrolysis inhibited in storage tanks may not be a hindrance on the economics of the whole system. *Urine bacterial communities during collection*

Biological samples for DNA sequencing were taken from the urinal-on-wheels and compared to the results from the glass urinal experiments. The glass urinal experiments were conducted in a laboratory and the glass urinal was autoclaved between runs to remove the influence of the restroom and the urinal-on-wheels plumbing on the bacterial communities. Fig. 4-5 shows the bacterial orders for the samples taken from Run 2 (no acid), Run 6 (least amount of acid added), and Run 8 (most amount of acid added) compared to samples from the glass urinal. Three glass urinal samples were sampled: a sample taken from the glass urinal trap after a urea hydrolysis experiment, a tank sample taken after a urea hydrolysis experiment, and a tank sample taken after an acid addition experiment. Results showing the pH and conductivity over time and the nutrient concentrations in the final samples can be seen in the SI (Fig. 4-S8 and 4-S9). The bacterial community results show that there was higher diversity in bacterial orders during experiments with no acid addition. When acid was added, a larger relative abundance can be classified as belonging to the Enterobacteriales order. The orders found in this study were in agreement with the orders of bacteria found in fresh urine samples in previous studies, even though the samples were taken after 2 to 3 weeks of urine collection (Lahr et al., 2016). This indicates that the bacterial communities had not begun to transition into the bacterial communities found in urine that has been stored for long storage periods. Lahr et al. found that pH and urine age were major drivers for changes bacterial community structures (Lahr et al., 2016). The urine pH in this study reached pH 8.9 at the end of Run 2 and was at pH 5.5 and 4.6 for Runs 6 and 8, respectively. Combined with the two week collection period, where fresh urine was continuously added to the tank, it can be concluded that the tank had not achieved the changes in pH and the storage time for a change in the community structure indicative of stored urine.



Figure 4-5. Bacterial communities based on 16S rRNA analysis for urinal-on-wheels Runs 2, 6, and 8 and glass urinal experiments.

This is the first study of its kind to sequence bacterial DNA from urine collected solely with nonwater urinals. There was a possibility that the bacterial community would not contain the enteric bacteria found in urine collected with urine-diverting toilets, where fecal contamination is a risk. However, that was not the case in this study, as seen
in Fig. 4-6. Only fifteen of the genera found in this study (n = 29) have been identified in previous studies on the urinary microbiome (Fouts et al., 2012; Hilt et al., 2014; Lewis et al., 2013; Nelson et al., 2010). Nine of the identified genera are characterized as gut microbiome bacteria. Specifically, *Escherichia* was one of the most abundant genera found in the urine samples from the urinal-on-wheels and the glass urinal experiments. The urine tanks without acid (Run 2 and Tank no acid in Fig. 4-8) had lower relative abundances of *Escherichia* compared to those with acidified urine, indicating that acetic acid addition selected for higher abundances of *Escherichia*. This difference in abundance may be due to the high pH conditions after urea hydrolysis, as E. coli survival decreases 100-fold at pH 8 (Small, Blankenhorn, Welty, Zinser, & Slonczewski, 1994). The presence of gut microbiome bacteria indicates that environmental factors played a role in introducing fecal contamination that was not expected when collecting urine with nonwater urinals alone. The urinal-on-wheels was placed in a real restroom, where aerosol droplets from toilet flushing could have deposited on the urinal surface. Flushometer-type toilets that are commonly used in CI buildings have been shown to produce higher numbers of droplets, which could carry bacteria and pathogens (Johnson et al., 2013). The benefit of reducing gut microbiota from urine collected with urinals alone was not realized in this study. Future research on implementation at a larger scale is needed to determine how the restroom setting contributes to the presence of fecal bacteria in urine collected with nonwater urinals.



Figure 4-6. Relative abundance of genera identified in Run 2, 6, and 8 and glass urinal experiments with and without acid addition.

Finally, as the urinal-on-wheels experiments served to inform future implementation of urine diversion in CI buildings, it is important to address the bacterial communities in terms of risk of exposure to those operating the system. An important implication is the presence of pathogenic bacteria found in the system, as it could have adverse effects on the staff operating the system (Fig. 4-7). With equal importance, while not being in the scope of this study, the presence of pathogenic viruses should also be an area of focus as urine diversion systems are implemented. Future research is need to determine the viability of the identified bacteria and viruses, as well as pathways for human exposure. The health and safety of the individuals is of paramount importance and their exposure the potentially pathogenic bacterial species identified in this study needs to be reduced (e.g., *Escherichia, Pseudomonas*). Bischel et al. (2019) followed workers in a container-based urine collection program and determined that the highest risk to workers existed during the collection process, where exposure to contaminated surface is higher, than during the resource recovery phase (Bischel, Caduff, Schindelholz, Kohn, & Julian, 2019). Therefore, with proper cleaning, personal protective equipment, and a closed design, a urine diversion system in a CI setting could reduce exposure to workers.



Figure 4-7. Relative abundance of species identified in Runs 2, 6, and 8 and glass urinal experiments with and without acid addition.

The engineering significance of identifying the bacterial species in the stored urine can be summarized by the following points. This is the first study to identify the bacterial communities in urine collected solely with nonwater urinals and from urinals in public restrooms, unlike other studies that identify bacterial communities in the medical literature or in systems using toilets for collection (Hilt et al., 2014; Lahr et al., 2016). Secondly, the results show that urine collected with nonwater urinals can still contain a wide range of bacteria, including pathogenic species. This is significant in terms of operation as it pertains to exposure to staff and in terms of the treatment processes used to recover urine-derived products. The novel results show that assumptions about collection with nonwater urinals alone cannot be made, in terms of bacterial community structure, and can be used to inform future implementation of urine diversion systems.

Urine chemistry during storage

The third and final objective of this work was to quantify the impact of acid addition on urine storage where fresh urine is no longer entering the system. Urine was treated with the three acid addition treatments and was stored at room temperature for 120 days. The results show that urea hydrolysis was still possible after acid addition, as seen by the pH, conductivity, ammonia, TN, TOC, and phosphate concentrations (Fig. 4-8). In general, the amount of acid added had an influence on the rate of hydrolysis in the storage bottles. The pH of Run 8 was held consistently at pH 5 for the 120 day storage period (Run 8 received the most amount of acid). The samples taken from the Run 8 storage bottle confirmed that urea hydrolysis was inhibited for the most amount of storage days. These results show that there is a possibility to control the speciation of nitrogen in storage tanks depending on the amount of acid added at the urinals thereby enabling urea recovery at pH 5 or ammonia recovery at pH 9. However, there are some exceptions to the assumption that amount of acid added influences rate of hydrolysis in storage. Even though Run 6 received the least amount of acid, it did not hydrolyze as rapidly as the run with the same control logic (Run 5). Similarly, Run 3 and Run 4 behaved differently in storage even though they received approximately 1% of acetic acid by volume of urine. Additionally, the results show that there was a discrepancy between

the ammonia and TN concentrations for Run 3, which is most likely due to error introduced by large dilution factors and instrumentation differences.



Figure 4-8. Trends in urine chemistry over storage time for urine from Runs 3–8. Error bars show the standard deviation for (c) – (f). The percent acid by volume of urine collected per run is displaying in the figure legend.

The results show that there is a relationship between acid addition at the urinals and the amount of urea hydrolysis inhibition in the storage tanks, making it possible to select for a system to recover either ammonia- or urea-nitrogen. For example, if the stakeholders design a system for urea recovery via forward osmosis (Hannah Ray et al., 2019), then additional acid addition would be needed in the storage tank if the amount of acid added at the urinals was not sufficient to inhibit urea hydrolysis in the storage tanks.

The urea recovery system could implement membrane separation for urea recovery and potassium-struvite precipitation for phosphate and potassium recovery (H. Ray et al., 2020; Xu et al., 2015). The ammonia recovery system could implement ammonia stripping, struvite precipitation for phosphate recovery, and distillation for potash and water recovery (N. Jagtap & T. H. Boyer, 2020). A limitation of this study was the shortened distance between the urinal and the urine tank on the urinal-on-wheels. In a real system, the collection pipes would expose urine to biofilms that could be rich in the urease enzyme and it could increase the rate of hydrolysis before the urine reaches the storage tank (N. S. Jagtap & T. H. Boyer, 2020; K. M. Udert, Larsen, Biebow, et al., 2003). This is especially important in multi-story CI buildings, as the distance between the urinals and the storage tank is longer than that in household urine diversion systems and where the collection system is connected to multiple urinals. The next step towards implementation of urine diversion would be to study acid addition for urea hydrolysis inhibition in a real multi-story CI building. The opportunity would bring the insight needed to operationalize a urine diversion system for nutrient recovery and water conservation.

Conclusions

A urinal-on-wheels was created that was able to monitor urea hydrolysis in real time and control the inhibition of the reaction using three different control logics based on historic Wifi login data. This paper was able to prove that Wifi login data can be used to inform the operation of urine diversion systems by approximating building occupancy and periods of increased urinal use. It was also the first study to identify bacterial communities for urine collected solely with a nonwater urinal and the results showed a large presence of gut microbiome bacteria that was not expected due to the avoidance of direct fecal contamination from the collection fixture. The shifts in community structure due to acid addition should be further investigated to determine increased risk to operators. Finally, acetic acid addition at the urinal has impacts on the rate of urea hydrolysis in storage tanks and can be used to select for urea- or ammonia-nitrogen depending on the nutrient recovery technology available or the targeted nutrient product of interest.

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CHAPTER 5

DATA MINING METHODS PREDICT CHLORINE RESIDUALS IN PREMISE PLUMBING USING LOW-COST SENSORS

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Abstract

Variable water quality within buildings is of increasing concern due to public health impacts (e.g., lead, *Legionella pneumophila, Naegleria fowleri*, disinfection byproducts). Advances in data acquisition and analytics provide the opportunity to monitor real-time building-wide water quality variability. Accordingly, the goal of this research was to create a water quality sensor platform including data acquisition, storage, and mining methods able to monitor and ultimately improve water quality within buildings. The platform was used to monitor water temperature, pH, conductivity, ORP, DO, and chlorine using sensors only. Other building data infrastructure, specifically Wifi logins by occupants, were used to approximate activity rates and associated water use. An advanced machine learning technique, gradient boosting machines, predicted the chlorine residuals throughout the building plumbing network better than multivariate linear regression models. Finally, the implications of water quality monitoring on costs, scalability, reliability, human dimensions, regulatory compliance, and future green building designs are considered.

Introduction

Recent incidences of drinking water related illnesses have led to an increased interest and need for research on the variable water quality found in buildings (i.e., premise plumbing) (Rosen, Pokhrel, & Weir, 2017). The development of low-cost monitoring technology has enabled researchers to study drinking water systems that were once difficult to monitor. Low-cost sensors and microcontrollers, in conjunction with the ease of access to cloud storage technology, has provided the opportunity for real-time monitoring and control of water quality at the point-of-use (Cloete et al., 2016; Rao et al., 2013). Water quality undergoes changes across water treatment plants and within both municipal distribution systems plus private home and institutional, or commercial premise plumbing systems such as hospitals, hotels, schools, etc. (Hull et al., 2017; National Research Council, 2007). Three water quality constituents of interest in premise plumbing are opportunistic pathogen outbreaks, such as Legionella pneumophila and *Naegleria fowleri*, disinfection byproducts (DBPs), such as trihalomethanes (THMs) and haloacetic acids (HAAs), and corrosion and leaching of metal pipe materials, such as lead and copper (Cope et al., 2015; Dion-Fortier, Rodriguez, Sérodes, & Proulx, 2009; Falkinham, 2015; Masters, Welter, & Edwards, 2016). Few premise plumbing systems track water quality throughout vast systems of piping and associated storage systems, despite emerging recognition that stagnation and building operations (e.g., water temperature) impact water quality. There is an opportunity to leverage the advancements in water quality sensor technology to address concerns in premise plumbing, especially related to the relationship between chlorine residual and Legionella and THMs. If strategically located throughout the building water system, a low-cost sensor network

would be able to provide real-time measurements of drinking water quality parameters with seasonal or spatial variability that would otherwise be unknown with periodic (e.g., quarterly) sampling.

This paper explores the use and implications of a water quality sensor platform to monitor and maintain water quality within premise plumbing as a tool in the effort to address the water quality conditions that are associated with the occurrence of *Legionella* outbreaks and elevated THM concentrations. Commercial and institutional (CI) building facilities mangers are tasked to maintain water systems within buildings while operating in a regulatory island where regulations are nonexistent and standards are enforced by local building codes; not the federal or state environmental agencies who enforce drinking water quality ("Standard 188-2018 -- Legionellosis: Risk Management for Building Water Systems (ANSI Approved)," 2018). The question arises, what will incentivize facilities managers and owners to install a water quality sensor platform in their building? We propose that pressure to install such a system can come from the response to awareness of two threats to water quality: (1) a response to acute threats (e.g., Legionella outbreaks) from risk assessors such as building insurers, and (2) a response to chronic dangers (e.g., lead or DBP exposure) from informed citizens. For example, prolonged shutdowns of institutional buildings associated with the COVID-19 pandemic and subsequent monitoring in schools is revealing detection of L. pneumophila among the types of high profile events that magnify even routine impacts of building operations (e.g., weekends) when building activity influence water quality (Horberry, 2020). A lowcost water quality sensor platform, such as the one designed, implemented, and tested in this study, would be able to monitor and predict drinking water in premise plumbing

systems in CI buildings to ultimately prevent both acute and chronic threats to water quality.

The goal of the research described in this paper was to create a proof-of-concept water quality sensor platform for data acquisition, storage, and mining methods able to monitor and inform the process of ultimately improving water quality within buildings, and to serve as a testbed to determine the minimum number of sensor types and locations needed to understand water quality in real-time. The water quality sensor platform focused on measuring and predicting chlorine residual because disinfectant concentration drives numerous chemical and microbiological processes that lead to undesirable outcomes (e.g., THM, lead and copper corrosion, biofilms, *Legionella*, tastes and odors), and chlorine sensors alone are costly to measure in real-time within premise plumbing (Berry, Xi, & Raskin, 2006; Cantor, Park, & Vaiyavatjamai, 2003; Chowdhury, Champagne, & McLellan, 2009; Dietrich & Burlingame, 2020). Using lower cost sensors (<\$300 per sensor) within the water quality sensor platform can be an alternative to the high-cost chlorine sensors (>\$2000 per sensor) available in the market, as will be discussed later in this paper. The specific objectives were to (1) create and implement a water quality sensor platform in a multi-story building, (2) assess the water quality patterns based on the data collected using water quality sensors, (3) model water quality using a traditional regression technique, (4) model water quality using a machine learning technique, and (5) discuss system considerations that are important for future implementation of water quality sensor platforms. The aim of the models is to design smart and responsive system using the minimum number of low-cost sensor types and sampling locations needed to improve water quality. The implications of implementing

water quality sensing in buildings are proposed in terms of capital and operational costs, scalability, reliability, human dimensions, and regulatory compliance.

Water Quality Sensor Platform

The water quality sensor platform used in this study was installed in a multi-story institutional building (i.e., office and laboratory building) at Arizona State University (Tempe, AZ). Interdisciplinary Science and Technology Building 4 (ISTB4) is a sevenstory building opened in May 2012. The building houses laboratory space for a range of scientific fields in approximately 300,000 sq.ft. About half the space is dedicated to offices for professors and graduate students. The 1st and 2nd floors house the lobby, K–12 educational spaces, and laboratory spaces. Excluding the 1st floor, each floor has a small breakroom containing a sink, a microwave, two refrigerators, coffee makers, and a small table. Domestic cold water (DCW) for ISTB4 is supplied by City of Tempe, Arizona. A 6 inch main starts at the basement with one riser to feed the breakrooms and bathrooms. The copper riser starts at 2 inch and becomes 1¹/₄ inch by the 7th floor. Floors have one breakroom and one alcove with bathrooms and two water fountains. Bathrooms are located vertically on top of each other for all floors and breakrooms start at the 2nd floor which are also vertically co-located (see floor plans in Fig. 5-S1).

To narrow the scope of this pilot sensor network in our testbed building, three floors were chosen for this study: 2nd, 3rd, and 7th. Six water quality sensors were installed underneath the breakroom sinks to monitor the water quality in real time. The following parameters were monitored for this study and the sensors were installed in series: pH (Atlas Scientific, ENV-40-pH), conductivity (Atlas Scientific, ENV-40-EC-K1.0), temperature (Atlas Scientific, PT-1000), dissolved oxygen (DO, Atlas Scientific, ENV- 40-DO), oxidation reduction potential (ORP, Atlas Scientific, ENV-40-ORP), and free chlorine (Chemtrol, PPM010) (see Fig. 5-1). The flow cell branches off the domestic cold water line via a 1/4 inch tee that runs through the flow cell continuously and exits to the sink drain pipe after an air gap placed for safety.



Figure 5-1. Architecture of the flow cell used in the water quality sensor platform

A Raspberry Pi 3 Model B was used as the system controller. Two Whitebox Tentacle T3 boards were stacked onto raspberry pi 3 Model B GPIO pin headers. The T3 boards were used to interface Atlas Scientific sensors without the need of wiring or breadboards. Each T3 board can accept up to 3 Atlas Scientific sensors and their corresponding trademarked circuits (Atlas Scientific EZO circuits) to handle the analog 107 to digital conversions. Each EZO circuit had a unique address that the Raspberry Pi used to call individual sensor one at a time to get a reading. When a reading was called for, the EZO circuit read the sensors' analog signal, scaled it, and converted it to a digital signal, which is read and stored by the calling program via the inter-integrated circuit (I^2C) communications bus. The free chlorine sensor is a 4 – 20 mA output sensor, therefore an MCP3008 analog-to-digital converter integrated circuit was used to convert the signal to a 10 bit digital signal that was read by the calling program via the serial peripheral bus (SPI) communications bus. All sensor readings were stored into a variable by the calling program and written to a local csv file once they are all collected. After being logged on the csv file, the program connected to the Google Sheets application programming interface (API) via Wifi connection and uploaded the values to the Google Sheet. Next, a connection was opened to the Adafruit IO API and the values are uploaded to the online dashboard. Measurements were taken serially for every sensor once every 5 minutes.

Wifi login data was used a surrogate data to aid in understanding activity within the building and the difference in activity on different floors of the building, which would impact spatial water use patterns within the building. In a novel approach, Wifi login data was used for this purpose because it is able to measure activity on each floor as occupants move throughout the space and their Wifi devices access the internet through access points located throughout the floors. The Wifi login data is collected by the ASU Information Technology (ASUIT) department in real-time. The Wifi login data was provided for this research as a sum of all Wifi logins that occurred at each hour across all access points for all seven floors in the building. Each Wifi login measurement represented a single login to a Wifi access point. An increase in Wifi logins is representative of increased activity on the floor level, as more Wifi devices are connecting via the access points. Previously, Wifi login data has been used in the literature as approximation for occupancy (Ghai, Thanayankizil, Seetharam, & Chakraborty, 2012) and most applications have been shown for control of HVAC systems in office buildings (W. Wang et al., 2019). Recently, Wifi logins have now been correlated to drinking water and wastewater systems within CI buildings (Richard et al., 2020; Saetta, Zheng, Leyva, & Boyer, 2020).

Data Analysis and Model Calibration

Data preprocessing

Data was preprocessed in order to create two types of models: (1) linear regression models and (2) gradient boosting machines. Data acquisition, modeling, and predictive parameterization was performed in the modeling language R and Google Sheet programs. The six water quality measurements per floor were taken every 5 min. Prior to creating the models, the data had to be prepared to merge the water quality sensor data and the Wifi login data, to remove outliers within the water quality sensor data, to remove blanks found in the data set, and to normalize the data set. The data was first entered into Microsoft Power BI to create easy-to-use queries for the data timeframes that were desired for the models. In Power BI, the following data sets were exported for data analysis in R: time stamps, pH, conductivity (μ S/cm), temperature (°C), free chlorine concentration (mg/L), ORP (mV), and DO concentration (mg/L).



Figure 5-2. Data flow schematic showing the steps taken to prepare "normalized data" for the linear regression models and "preprocessed data" for the GBM models.

Four steps were taken to preprocess the data before models were calculated (see Fig. 5-2). First, in order to match the 5 min water quality data points to the hourly Wifi data points, hourly averages of each water quality parameter were calculated in order to join the water quality data set with the Wifi login data set. Second, outliers were defined as any point outside the whiskers of a box and whisker plot. The outliers were removed from each water quality parameter in any case where they were identified. Outlier removal is an area of research and many forms of identification and removal of outliers are tested in the literature (Phan et al., 2020). The method for identification and removal of outliers in this paper were chosen for their low complexity but it is acknowledged that more robust methods for treating outliers could have been used. The third step was to remove any data rows that contained an NA value. NA values occurred during points when the sensors were not operating correctly or during periods where Wifi login data

was not available. At this point, this data became known as the "preprocessed data" because it was preprocessed (averaged, removal of outliers, etc.), but it was not normalized. The preprocessed data was then normalized using the min-max scaling method, where the data values were normalized between values of 0 and 1 (data known as "normalized data", see Fig. 5-S2, 5-S3, and 5-S4 for density plots and Pearson correlation coefficients of the normalized data).

In R, Welch's t-tests were used to compare the means of the water quality parameters and the Wifi logins for each floor (Welch, 1947). Welch's t-tests were chosen because they allow for sample groups of varied lengths and unequal variances, as opposed to Student's t-tests or ANOVA analysis (Delacre, Lakens, & Leys, 2017). Three pairs of tests were run: (1) comparing the 2^{nd} and 3^{rd} floors, (2) comparing the 2^{nd} and 7^{th} floors, and (3) comparing the 3^{rd} and 7^{th} floors. A 95% confidence interval (p-value < 0.05) was used for all t-tests to determine statistical significance.

Linear regression models

The normalized data were used to make multiple linear regression models of the free chlorine concentration in premise plumbing. First, the *ggpairs* function in R was used to calculate the correlation coefficients for all pairs of data parameters and the *leaps* function in R was used to search for the best fit among parameter subsets for linear regression. The functions were used to determine which subset of parameters (i.e., pH, conductivity, temperature, ORP, and DO) were best for modeling the free chlorine concentration. Next, linear regression models were created on a variety of parameter pairs based on the results.

The linear regression models were trained using 70% of the total data set and they were tested with the remaining 30%. Three models per floor were trained and tested: (1) a model using only the Wifi login data [Chlorine (mg/L) = $a + (b \times Wifi)$], (2) a model using only the ORP and Wifi login data [Chlorine (mg/L) = $a + (b \times Wifi) + (c \times ORP)$], and (3) a model using all available data [Chlorine (mg/L) = $a + (b \times Wifi) + (c \times ORP)$] + ($d \times pH$) + ($e \times Cond$) + ($f \times Temp$) + ($g \times DO$)]. The models' statistics were returned by the R lm function. The adjusted R² values were used to compare the model fit. Using the testing data, the models were used to predict the chlorine concentration and the root mean square error was calculated for the actual data (testing data set) and the predicted model data. The predicted and actual values were compared using a Welch's t-test (95% confidence interval, p-value < 0.05) to determine if there was a statistically significant difference between the two groups.

Gradient boosting machines (GBMs)

The preprocessed data was used to make three gradient boosting machine models to predict the free chlorine concentration in the premise plumbing. The UC Business Analytics R Programming Guide Gradient Boosting Machines (GBM) tutorial was used as a guide to create the models used in this paper (Boehmke & Greenwell, 2019). The GBMs were trained with 70% of the total data set and tested using the remaining 30%. Models for each floor were trained and tested to predict free chlorine concentration using (1) the Wifi logins and ORP data, (2) all parameters (Wifi logins, ORP, pH, conductivity, temperature, and DO) and (3) data for the top two parameters identified by the relative importance results from the all-inclusive models. The R GMB package was used to train a model with default parameters (0.001 shrinkage rate, 1 tree depth, and 5-fold crossvalidation). The cross-validation RMSE (Root Mean Square Error) given by this initial model was a close approximation of the RMSE that could be achieved with GBMs. Next, a tuning method was used to determine the best model parameters for the data sets. Eighty-one different combinations of the shrinkage rate, tree depth, minimum number of observations at the tree nodes, and the fraction of training data subsampling to introduce stochastic gradient descent were created to train a tuning model and to determine the best set of parameters. The tuning models were run multiple times to determine the best combination of parameters that resulted in the lowest RMSE. The final parameters can be seen in Table 5-S1. Once the final models were made, the parameter relative importance was calculated to determine the influence each parameter had on the model (this was used to choose parameters for the third type of GBM models). The final models were then used to predict the free chlorine concentration for each floor. The RMSE of the predicted versus the actual data was calculated to compare across models. The predicted values were also compared to the actual values using Welch's t-test (95% confidence interval, pvalue < 0.05) to determine if there was a statistically significant difference.

Results and Discussion

Observed trends in water quality

This is the first study of its kind to measure water quality parameters in institutional building premise plumbing in real-time with low-cost online sensors alone. Figure 5-3 summarizes the water quality trends observed in the preprocessed data for the six water quality sensors used in the water quality sensor platform (data points for the boxplots are listed in Table 5-S2). Three statistically significant trends were shown by the data as floor levels increase within the building: (1) temperature increases, (2) free

chlorine residual decreases, and (3) ORP decreases (p-values shown in Table 5-S3). More specifically, the mean temperature increased from 21.5°C to 25.3°C, the mean chlorine concentration decreased from 0.43 mg/L to 0.01 mg/L, and the ORP decreased from 724 mV to 268 mV from the 2nd floor to the 7th floor. These trends are reasonable based on physical and chemical processes and have implications as they pertain to DBP formation and microbial risk. Namely, relationships are well known to exist between residence time or temperature with chlorine decay, or between ORP measurements and chlorine concentrations (Copeland & Lytle, 2014; Ozdemir & Buyruk, 2018; Powell, Hallam, West, Forster, & Simms, 2000). The increase in temperature is mirrored by a decrease in chlorine concentration, which has negative implications on the water quality. Values for the other three parameters (pH, conductivity, and DO) do not show trends based on floor level. Conductivity is a measurement of salt content of the water, and since no chemicals are being added the lack of change in conductivity was expected but served as a negative control to detect potential anomalies (e.g., cross connections with whole-building ultrapure water systems, etc.). Likewise, pH was not expected to vary within the building mainly because the local tap water (Tempe, AZ) is well buffered by its high alkalinity (average 175 mgCaCO3/L; range 130 to 370 mgCaCO3/L)). However, in other communities with lower alkalinity in tap water, tracking pH could be a useful analyte because it could have an influence on corrosion of heavy metals (Edwards, Ferguson, & Reiber, 1994; Reyes, Letelier, De la Iglesia, González, & Lagos, 2008). Conductivity and pH can vary seasonally entering the building from the municipal water supply, and can be useful to understand or identify influences of different water sources (e.g., in-distribution system blending of multiple surface water treatment plants, groundwater pumping,

purchased water across different pressure zones) used by municipalities on individual building water quality. DO concentrations for the 7th floor were not included in this data mining because of sensor error identified during data preprocessing. A majority of the DO concentrations were significantly higher than the theoretical maximum of DO in water at these conditions (see Fig. 5-S5). This points to the importance of review of data trends by experts in water quality, especially when implementing in building locations with low chlorine due to the physical constraints of the system (in our system, the 7th floor is the highest floor level and inherently, chlorine concentrations were low). Experts may also determine that the absolute values of the measurements are not important when systems are designed to identify long-term trends in the water quality data.



Figure 5-3. Boxplots for the preprocessed water quality for the 2^{nd} , 3^{rd} , and 7^{th} floors prior to normalization. The interquartile ranges (IQR) between the 25^{th} (Q₁) and 75^{th} (Q₃) quartile and the whiskers extend to Q₁ – 1.5×IQR and Q₃ + 1.5×IQR. The center line represents the median (Q₂) of the dataset. Outliers are shown beyond the whiskers.

Welch's t-tests were used to compare the means of each pair for the six measured parameters (2nd vs 3rd floor, 2nd vs 7th floor, and 3rd vs 7th floor). The resulting p-values (see Table 5-S3) indicate that there was a statistically significant difference between all pairs except for conductivity values between the 2nd and 3rd floor. There was a statistical difference in the measured parameters between floors, even when there was no apparent increasing or decreasing trend among individual parameter values (pH, conductivity, and DO data). These results also demonstrate that the water quality sensor platform was able to capture differences in water quality from floor to floor within the multi-story building that would otherwise be difficult to capture with grab samples. Thus, the water quality sensor platform provided a data set that is far richer than what is possible with traditional sampling techniques. The challenge is to determine the number and placement of water quality sensors necessary to improve building water quality for its occupants. *Relationships observed in water quality and building biometric datasets*

The in-line sensor water quality data were augmented with nearly continuous Wifi login data which served as our biometric surrogate—building occupant behavioral patterns by floor in the building. Figure 5-4 shows the relationship between Wifi logins, accumulated on an hourly basis, and the six water quality parameters. Linear regression lines are shown to provide insight into the trends observed in the data (see Table 5-S3 for adjusted R² values of the relationships). Two parameters (chlorine residual and conductivity) had an increasing trend as Wifi logins increased. The chlorine residual is expected to increase as Wifi logins increase because more people would be drawing water into the premise plumbing system from the distribution system, where chlorine residual is higher (Richard et al., 2020). From Figure 5-3, conductivity values do not depend upon floor level, and the apparent correlation is therefore unexpected. Unexpected correlations are common when analyzing big data sets. This is when it becomes important to use expert judgement in interpreting the results, as it can become evident that the statistical relationship does not always indicate causation between two parameters, such as the results showing a relationship between Wifi logins and conductivity.

The other four parameters (ORP, pH, DO, and temperature) have more complex relationships with Wifi usage patterns, including when one floor level has the opposite relationship to Wifi logins compared to the remaining two floor levels. ORP had a negative relationship with increasing floor level (see Fig. 5-3) while a positive relationship was observed with increasing Wifi logins on the 3rd and 7th floor (see Fig. 5-4). This agrees with the relationship between chlorine and ORP shown in the literature (Copeland & Lytle, 2014). A recently published study was able to correlate the Wifi logins to water use within a university building with a correlation factor as high as 0.74 (Richard et al., 2020). This creates the opportunity for future research to confirm whether Wifi logins could be used to approximate water use within a variety of building types as opposed to relying on costly submetering of flowmeters or other direct measurements. Using Wifi login data may provide beneficial information with a level of acceptable accuracy, without the high cost involved with installing location-specific water meters. There may be other biometric data surrogates actively collected within buildings (e.g., security cameras, elevator use, temperature sensors, thermostats, motion detectors installed to activate lighting, etc.) for reasons unrelated to water use patterns, but which could be harnessed to generate high resolution water use patterns in buildings.



Figure 5-4. Wifi logins versus the six water quality parameters. Trendlines are shown for each floor.

Multivariate linear regression models

The traditional approach in water quality modeling is to create multivariate linear regression models (B. Chen & Westerhoff, 2010; Ged, Chadik, & Boyer, 2015; Lu, Krasner, & Liang, 2011). The multivariate linear regression models developed in this research to predict chlorine concentration show low predictive capability as indicated by the low high RMSE between the actual and predicted values. However, the models are discussed here for completeness to compare with advanced modeling technique used in section 4.4. Table 5-1 summarizes the multivariate linear regression models were based on three

different groupings of parameters: (1) Wifi logins only, (2) Wifi logins and ORP, and (3) Wifi logins, ORP, pH, conductivity, temperature, and DO. The second model type was selected to explore the possibility of using one low-cost sensor in addition to Wifi logins to predict chlorine concentrations. ORP was chosen for this case because of its known connection to chlorine concentration; Wifi logins are related to water age via building activity and ORP is related to chlorine concentrations (Copeland & Lytle, 2014; Richard et al., 2020). The Wifi logins and ORP models represent models made with covariates that have a known relationship with chlorine. The coefficients for each grouping of parameters are shown in Table 5-1. A few trends can be seen in the model coefficients: (1) Wifi logins and ORP have a positive relationship with chlorine concentration, (2) temperature has an inverse relationship with chlorine concentration, and (3) pH, conductivity, and DO do not influence the models in the same manner from floor to floor. In terms of predictive capability, the adjusted R² values suggest that the models for the 3rd floor were the best in predicting the chlorine concentration.

Model		Model parameters and coefficients							Statistical analysis		
#	Floor	а	b	С	d	е	f	g	Adj.	RMS	T-
'n		(int.)	(Wifi)	(ORP)	(pH)	(cond)	(temp)	(DO)	R ²	Е	test
1	2	0.52	0.05	-	_	-	-	-	4.3E-04	0.22	0.58
2	2	0.10	0.08	0.49	-	-	-	-	0.12	0.21	0.93
3	2	0.15	0.09	0.36	0.17	0.25	-0.28	-0.09	0.15	0.21	0.98
4	3	0.11	1.08	-	-	-	-	-	0.43	0.18	0.14
5	3	-0.07	0.93	0.43	-	-	-	-	0.59	0.16	0.64
6	3	-0.19	0.82	0.32	0.21	-0.15	-0.25	0.29	0.69	0.14	0.57
7	7	0.05	0.41	-	-	-	-	-	0.07	0.19	0.24
8	7	-0.02	0.35	0.18	-	-	-	-	0.12	0.18	0.27
9	7	0.23	0.39	0.14	-0.29	0.003	-0.14	-	0.14	0.18	0.18

Table 5-1. Summary table for linear regression modeling results and statistical analysis. Model equation: Free Chlorine = a + Wifi × b + ORP × c + pH × d + Conductivity × e + Temperature × f + DO × g.

Once the models were trained, they were used to predict chlorine concentrations using the testing dataset. T-tests were run on the predicted values versus the actual values with a significance level of p < 0.05. The t-test results show that there was no significant difference between the predicted and actual values for all models, which is the desired result. Figure 5-5 shows the predicted versus actual chlorine concentrations using the Wifi login and ORP data models (models 2, 5, 8) from Table 5-1 (see SI (Appendix C) for figures showing results of remaining models). The linear regression model for the 3rd floor was able to predict the values to a greater extent than the models for the 2nd and 7th floors, based on the adjusted R² > 0.5 as opposed to the other models. Additionally, the calculated RMSE for the 2nd and 7th floors show that including an increased number of parameters in the model did not decrease the RMSE from the models using Wifi logins alone. The only considerable decrease in RMSE as more parameters were added to the model was seen with the models for the 3^{rd} floor (models 4, 5, 6).

The multivariate linear regression models were not able to fully capture the trends provided by the data. The adjusted R² values for the models using all water quality parameters and the Wifi data was only able to reach 0.16 and 0.14 for the 3rd and 7th floors, respectively. There is an apparent ceiling effect occurring in the predicted values in Figure 5-5, where the predicted values do not increase past an apparent asymptote at 0.5 mg/L chlorine. Based on the data gathered for this system, it is evident that linear regression models were not able to model the chlorine concentration, even when all sensors and data sources were used.



Figure 5-5. Linear regression modeling data versus measured data for models using Wifi logins and ORP data only (models 2, 5, and 8). The 1:1 line is displaying on each graph. Chlorine (mg/L) = $0.10 + (0.08 \times \text{Wifi}) + (0.49 \times \text{ORP})$ Chlorine (mg/L) = $-0.07 + (0.93 \times \text{Wifi}) + (0.43 \times \text{ORP})$ Chlorine (mg/L) = $-0.02 + (0.35 \times \text{Wifi}) + (0.18 \times \text{ORP})$

Gradient boosting machines (GBMs)

An advanced machine learning method achieved better prediction than the multivariate linear regression models. GBMs were chosen in this case because they provide a robust method that can be used to model systems without the need for data preprocessing (Natekin & Knoll, 2013). This method can produce models with low RMSE by using methods to reduce overfitting (i.e., when a model is too specific to the

training data and cannot be used to generalize across different data sets). A GBM is built by sequentially training weak, small models that learn from error of the previously trained models (i.e., an ensemble of models). Three GBM models were created: (1) models using Wifi login and ORP data, (2) all-inclusive models using all available water quality parameters, and (3) models using top two water quality parameters identified from relative importance results of the all-inclusive models. Similarly to the linear regression models, the first GBM was made to show the capability of a GBM model using one low-cost sensor in addition to Wifi logins to explore a system with a sensor pair that has a known relationship with chlorine. The third type of models was created by allowing the data to determine the two parameters for prediction, in order to keep complexity low while using the data to select the explanatory water quality parameters. Table 5-S1 shows the model parameters used in the final GBMs. Each parameter value was selected using the matrix method for model tuning and the model parameter values resulting in the lowest RSME were chosen.

Once the final models were trained, the water quality parameter relative importance was calculated for each model (see Table 5-2). Each floor has a different trend of water quality parameters of high to low importance, which points to each floor having unique characteristics. This could be caused by differences in activity and water use patterns, or hydraulic plumbing differences among the building floor levels. Based on the results, it cannot be concluded that a certain sensor cluster (i.e., water quality parameter) can be used to predict the chlorine concentration in premise plumbing across a variety of settings (i.e., floor level, sampling node). However, the results can be used to create unique models for each floor using the water quality parameters that had the

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highest importance. The models using the top two parameters (models 12, 15, and 18) were able to reduce the RMSE of predicting chlorine concentration when compared to Wifi logins and ORP concentrations, the models made with prior knowledge of water systems (models 10, 13, and 16). Based on the relative importance of variables in the all-inclusive models, the following water quality parameters were chosen for the third type of GMB models: (1) conductivity and DO, (2) temperature and Wifi logins, and (3) ORP and temperature for the 2nd, 3rd, and 7th floors, respectively.

Model	Eleor		R	Statistical analysis					
#		Wifi	ORP	рН	Cond.	Temp.	DO	RMSE	T-test
10	2	22.6	77.4	-	_	_	-	0.15	0.69
11	2	3.6	21.5	8.5	27.1	16.2	23.1	0.09	0.61
12	2	_	_	_	59.3	_	40.7	0.13	0.69
13	3	68.9	31.1	-	-	-	-	0.10	0.50
14	3	28.2	7.6	5.5	7.0	38.4	13.2	0.06	0.83
15	3	37.9	-	-	-	62.1	-	0.09	0.86
16	7	28.6	71.4	-	-	-	-	0.01	0.53
17	7	20.5	34.2	13.1	9.7	22.6	-	0.01	0.68
18	7	-	57.4	-	-	42.6	-	0.01	0.29

Table 5-2. Summary table of gradient boosting machine modeling results and statistical analysis.



Figure 5-6. Gradient boosting machine modeling data versus measured data for (A) models using Wifi logins and ORP data only (squares; models 10, 13, and 16), (B) for models using all parameters (circles; models 11, 14, and 17), and (C) for the models using the top two water quality parameters identified by relative importance for the all-inclusive GBMs (triangles; models 12, 15, 18). The 1:1 line is displaying on each graph.

Table 5-2 summarizes the results for the statistical analysis of the GBMs. All nine models had lower RMSE values than their multivariate linear regression counterparts. Figure 5-6 shows the predicted versus actual chlorine concentrations for the GBM models. The t-test results (p < 0.05) show that there was no significant difference in the predicted versus actual data for all nine GBM models. When comparing the RMSEs, the GBMs were able to reduce the RMSEs by half or more from those found with the multivariate linear regression models. In all cases, the RMSE of the GBMs was reduced by the addition of more model parameters. Overall, these results show that the sensors

used in this study could be used to predict chlorine by using GBM model. The resulting predictions do not show the ceiling effect on chlorine concentration that was seen with the linear regression models (see Figure 5-5). Based on the lower RMSE and the t-tests, the GBMs resulted in better predictions than the multivariate linear regression models for all three floors. And based on the current understanding of water chemistry in premise plumbing, it can be concluded that there is an opportunity to apply GBM-type models as prediction tools for building water systems. Future research is needed to determine the accuracy of these models during application and their ability to inform the improvement of water quality in building water systems.

While prediction was better for the GBMs, the prediction of the GBM models could be further improved by: (1) improving the raw data quality, (2) including a data source that was not included in these models, or (3) by including more raw data. The model performance is highly dependent on the accuracy of the sensor measurements. Ensure that the sensors are calibrated and operating correctly can aid in improving the raw data. The malfunctioning DO sensor on the 7th floor is an example of how a malfunctioning sensor can be incorporated into a model if expert knowledge about the physical limits of DO in drinking water. The model prediction can also be improved by adding explanatory data sources that were not including in the models. For example, the 7th floor models had low predictive capability due to the low concentrations of chlorine measured on that floor. Chlorine was expected to be low in the highest floor levels due to increased water age (William J. Rhoads et al., 2016). Is there a data source that could be used to account for the physical constraints of chlorine concentrations on the highest floor levels? These new data sources could be added to the model to improve prediction

where chlorine is limited by a spatial element. Finally, the models could be improved by including more raw data. The initial observation period can be longer to include more measurements in the training data. The treatment of outliers can also be altered to determine if more data points can be included in the models. In the end, this paper serves as a proof-of-concept in using GBMs to predict water quality in building water systems and future implementation of these machine learning models can be used to tune the models for better prediction.

System considerations

Advances in sensing technology has increased the number and variety of products available to monitor water quality in real time, with the development of a wide variety of sensors targeting parameters of interest pertaining to water quality. The capital costs of individual sensors can be still be prohibitive in their widespread application. Table 5-3 shows the cost of each sensor used in this water quality sensor platform and the total cost is representative of the cost of one sensing node within the platform. As is evident by the cost, installing a chlorine sensor at multiple places within a building can easily become very costly. More data, from more sensors may not be necessary – as we have shown through machine learning correlations in prior sections. The models in this paper were created to predict chlorine concentration data in order to reduce the dependence on the high-cost chlorine sensor by using a low-cost suite of sensors in its place.

	, +
ORP, Atlas Scientific	192
Conductivity, Atlas Scientific	215
Temperature, Atlas Scientific	68
Dissolved Oxygen, Atlas Scientific	283
pH, Atlas Scientific	164
Free Chlorine, Chemtrol	2134
Wifi login data	0
Total cost	3056

Table 5-3. Cost of each water quality sensor used in the water quality sensor platform.

The activity data, via the Wifi login data, provides the opportunity to include a human dimension into the models while not increasing the capital cost. The model results show that it may be viable to create models solely using Wifi login data, or other forms of measuring building activity, when including additional sensor data does not significantly reduce the RMSE of the models. Figure 5-7 shows the relationship of cost and RMSE for the models created in this study. Based on the results, the GBM models using Wifi logins and ORP data were able to decrease the RMSE of using Wifi login data alone while not increasing the cost of each sensing node in the platform. The GBMs using all parameters achieved the lowest RMSE but it increased the cost of each node by almost 5 times. This cost difference is expected to become prohibitive across multiple sensing nodes, floors, and buildings, which calls to question whether more sensors would be implemented to improve the RMSE by a few percentage points. The third type of GBMs (using the top two parameters of importance) did increase the cost for floors 2 and 7, due to the fact that they did not use the no-cost Wifi login data. However, these pairs of sensors were able to reduce the RMSE while not increasing the cost as much as the all-inclusive models. On

the 3rd floor, the sensor pairs for the third type of GBM had a lower cost than the Wifi logins and ORP model, while also reducing the RMSE. The third type of model could become an option for those seeking to improve predictive capability while not increasing cost or complexity of the system by including all six data sources. In these cases, it is important to determine if the identified pairs of parameters truly have a relationship with chlorine concentrations or if they are improving predictive ability due to unknown data characteristics. Expert analysis and future research can help in answering whether the identified relationships are due to water chemistry and use patterns or if the relationships found in this study are unique to this system.



Figure 5-7. Sensor cost for one measurement node versus root mean square error for the five type of models created with the water quality data and the Wifi logins. The colors denote the floors and the shape denote the model types.

The final objective of this work was to provide a perspective on the future implementation of such systems on the operation of building water systems. A low-cost, water quality sensor platform has the potential to transform the current understanding of and ability to improve water quality within buildings, and on drinking water infrastructure as it pertains to capital costs, installation, operation and maintenance, scaling, human dimension, reliability, and regulatory compliance (see Fig. 5-8). It is important to account for these factors since the water quality sensor platforms would become part of critical infrastructure that delivers drinking water to the community. Therefore, a thorough analysis of the implications of sensors on building water systems is needed to determine gaps in knowledge towards implementation.



Figure 5-8. Implications brought on to drinking water infrastructure by a water quality sensor platform.
Conclusions

The low-cost water quality sensor platform was able to predict chlorine concentrations in the premise plumbing using Wifi login data and ORP measured in realtime. Novel use of machine learning, in the form of GMBs, was able to predict water quality better than the traditional linear regression models used in water quality modeling. Implementation of a water quality sensor platform in buildings should consider (1) capital costs, (2) installation, operation, and maintenance, (3) scalability, (4) human dimension, (5) reliability, and (6) regulatory compliance. The next steps for a water quality sensor platform include the consolidation of data from multiple sampling nodes and buildings onto one easy-to-use dashboard, which has been augmented to include the modeling capabilities described in this paper. Innovation within the water sector to include water quality technologies will likely be slow and face challenges; but being proactive and building collaboration with early building adopters could help utilities increase confidence among residents while also addressing public health and infrastructure resilience.

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CHAPTER 6

CONCLUSIONS, FUTURE WORK, AND INCREMENTAL STEPS TOWARDS RADICAL CHANGE

Conclusions

Urine diversion is the process by which human urine is collected at the source due to its inherent qualities. Urine diversion has many benefits, especially as they pertain to water conservation, nutrient recovery, and pharmaceutical removal, making it a viable technological process for future smart cities. This dissertation sought to understand urine chemistry and the urea hydrolysis reaction at the point of collection as a way of advancing the implementation of urine diversion systems in the United States. Each chapter of this dissertation was designed as an incremental step in the research process, from the testbed to restroom and towards operationalizing urine diversion systems. The following conclusions were drawn from the entirety of the work.

The main conclusion of this work is highlighted by the efficacy of acetic acid addition as a method for urea hydrolysis inhibition in nonwater urinals. Chapters 2, 3 and 4 have shown that acetic acid addition is able to inhibit urea hydrolysis in synthetic and real urine for the period of collection. This was shown by the low ammonia concentrations in samples taken throughout collection during experiments in the urinal testbed experiments and the urinal-on-wheels experiments. Acetic acid addition can be used in one of three ways: (1) as preventative maintenance for nonwater urinals in conventional systems collecting urine for wastewater treatments, (2) as urea hydrolysis inhibition for subsequent urea recovery, or (3) as urea hydrolysis inhibition for ammonia recovery (see figure 6-1). The ability to select for urea or ammonia recovery comes from the fact that acetic acid addition allowed for reversible urea hydrolysis inhibition, meaning that urea hydrolysis was shown to progress unaided in urine storage tanks after storage time. In general, adding more acid in the urinals led to longer storage times needed for urea hydrolysis, which allows for time to employ a technology for urea recovery, and adding less acid in the urinals created conditions for urea hydrolysis to progress more rapidly. This is a major break-through because it opens the opportunity for ammonia recovery post urea hydrolysis inhibition. Hence, acetic acid addition can be tailored to create favorable conditions for urea or ammonia recovery depending on the stakeholder needs and the available technology.





A second conclusion from this work comes from the analysis of the bacterial community structures after acetic acid addition. Acetic acid is a simple carbon that can serve as a carbon source for bacterial growth. This becomes concerning because bacterial growth can uptake nutrients that urine diversion systems seek to recover. Bacterial growth can also be concerning for maintenance staff health and safety, especially if pathogenic species are present in the stored urine. Therefore, the analysis of the bacterial community structures can be used to determine if the bacterial growth is impacting nutrient recovery or if systems have to be designed in a manner that reduces risk to operators. This analysis was also able to determine possible sources of the bacteria making up the community structure. Specifically, chapter 4 found that the relative abundance of *E. coli* was higher in urine with acid addition than the urine without acid addition. Identifying a large abundance of *E. coli* in this work was not expected, as it was assumed that urine collection with nonwater urinals alone would reduce the presence of bacteria with enteric origin. This was the first study of its kind to analyze the bacterial communities of urine collected solely with nonwater urinals. The presence of enteric bacteria can be attributed to influence from the restroom ecosystem, especially the influence of aerosols created by powerful flushometers in nearby toilets. These findings can be used to inform the implementation of urine diversion systems by providing evidence of a shift in the bacterial communities in which there could be increased risk to operators.

This dissertation used CPS technology as a tool in answering the research questions. Chapters 2, 3, and 4 answered research questions focused on urine diversion systems by using CPSs that allowed for real time monitoring and control of urea hydrolysis. Using the CPS created for the urinal testbed led to the ability to test restroom conditions that lead to increased rates of urea hydrolysis in the collection system. With those results, the urinal-on-wheels control logics that were tested showed that a reduced acid addition, i.e. only adding acid at points of high occupancy, was able to reduce the amount of acid added while still inhibiting urea hydrolysis. Chapter 5 used a CPS used to monitor drinking water quality in the premise plumbing of a green CI building. Similar to the urine diversion CPS created in the earlier chapters, this CPS relied on low-cost water quality sensors to monitor water quality at three locations within the building in real-time. Advanced data mining techniques were used to predict chlorine concentrations in the system. This chapter provides a demonstration of the type of research that can be conducted with data collected with CPS technology. In this context, the data mining techniques were used on water quality data, but they can be easily translated to other building water and wastewater systems. The future implementation of urine diversion systems and on-site nutrient recovery operations should rely on cyber tools to ensure that systems are run efficiently and that all system components are operating correctly. Data mining should be used to predict conditions that increase functionality, decrease nutrient losses, and reduces waste of system inputs (i.e, chemicals and energy). Figure 6-2 shows the major components of this dissertation. The addition of Chapter 5 completes this dissertation and provides a systematic understanding of the relationship between urea hydrolysis in urine diversion systems and the capabilities of cyber-physical systems.



Figure 6-2. Major components of this dissertation. Chapter 2 and 3 created and used a cyber-physical system called the urinal testbed to monitor and control urea hydrolysis. Chapter 4 advanced findings from the urinal testbed to monitor and control real urine in a real restroom using a urinal-on-wheels. This study found that the structure of bacterial communities was altered after acetic acid addition. Additionally, Chapter 4 proposed the acetic acid addition process as a method for tailored nutrient recovery, i.e. urea recovery or ammonia recovery based on the amount of acid added during collection. Finally, Chapter 5 provided a demonstration of the data mining that can be conducted with CPS

technology to better understand conditions within water and wastewater systems in the built environment.

Future Work

Although the Covid-19 pandemic paused work meant for this dissertation, a significant amount of planning was conducted to test the acid addition and cyber elements of this dissertation in a full-scale urine diversion system in Biodesign C at Arizona State University. Additionally, a survey focused on facilities managers was planned to determine their level of acceptance for urine diversion systems. The following paragraphs will document the planning phases for these two studies and will propose future research based on these planned projects.

Biodesign C is a five story building on the Arizona State University campus. The urinals on the top 4 floors are plumbed separately from the restroom waste lines. The urine pipe is then intersected on the ground level, where a tank can collect 30 gal (113 L) of urine. A CPS was created that would add acetic acid to each urinal using predetermined control logics and monitor urine chemistry in the urine storage tank. The goal of the study was to define the operation of a urine diversion system with urea hydrolysis inhibition in a CI building in the United States. The predetermined acid addition control logics were created based on the urinal-on-wheels results: (1) acid addition after every urinal event, (2) acid addition during periods of high occupancy activity, and (3) acid addition during periods of low occupancy activity. The occupancy would have been the final phase of this research, which has gone from proof-of-concept with the initial study on acid addition in nonwater urinals published in 2017 to the urinal

testbed study published in 2019 to the urinal-on-wheels study published in 2020. With each study, the scale increased, automation and control was improved, and characteristics of the study were enhanced, such as study location (lab vs real restroom) and urine matrix (synthetic vs real). Ultimately, this study and future research on urine diversion systems in the United States can fill the gaps in knowledge that exist for the application of these systems in the US setting.

Urine diversion systems have been mostly applied in European countries, with Sweden leading the field in number of full-scale applications. According to McConville, et at, 2017, the largest contribution of publications (16%) from 1995–2015 with the key words "urine diversion" were published by Swedish researchers (McConville et al., 2017). Therefore, a large portion of the implicit and explicit knowledge on large-scale urine diversion systems exists in Swedish literature and scientific community. Publishing on systems based in the United States can answer questions related specifically to challenges and opportunities that can only be faced in this context at full-scale. The Biodesign C urine diversion system could become a testbed for urine-based technologies, from the collection to the treatment, recovery of nutrients and products, and removal of micropollutants. And finally, having an operational urine diversion system on the ASU campus can serve as an educational tool for the community, based on water conservation, nutrient sustainability, and the connections between water and wastewater in the built environment.

The second planned study was a case study on the level of approval for urine diversion systems that exists within the facilities management community. Seven studies have shown high support and acceptance for urine diversion by users (>60% approval for

those surveyed) (Blume & Winker, 2011; S. K. L. Ishii & Boyer, 2016; Lamichhane & Babcock, 2013; Lienert & Larsen, 2010; Medilanski, Chuan, Mosler, Schertenleib, & Larsen, 2006; Pahl-Wostl, Schonborn, Willi, Muncke, & Larsen, 2003; Simha, Lalander, et al., 2018). However, across the studies, most have focused on user perception of urinediverting toilets and most have only focused on the users of the systems, not those in charge of maintaining the systems. A recent study has surveyed perceptions outside of the general user population group. Simha, et al. surveyed farmers on their attitudes towards urine-derived fertilizers and found that 59% of farmers held a positive opinion of urinederived fertilizers (Simha, Lalander, Vinnerås, & Ganesapillai, 2017). Similarly, Medilanski, et al. interviewed 34 decision-makers in China who identified 20 years in the future as adequate time for increased implementation of separation of urine (Medilanski et al., 2006). While studies have identified social barriers, such as having to dispose of soiled toilet paper in a trash bin, as barriers towards acceptance in the general population (S. K. L. Ishii & Boyer, 2016; Lienert & Larsen, 2010), there has not been a study on the managerial barriers that may arise from implementation of urine diversion systems. The closest conclusions that involve management of these systems are seen in papers that describe the difficulty of attaining and retaining pathways to agriculture operations that are willing to use the collected urine or the urine-derived fertilizers (Berndtsson, 2006). Therefore, this study would fill a gap in the literature by focusing on the humans that will manage these systems in CI buildings.

Prior to the Covid-19 pandemic, and IRB approval had been acquired to conduct this study. The survey and semi-structured interviews would be conducted using the new ecological paradigm and open-ended questions that would allow for open discussion on the topic (Dunlap, Van Liere, Mertig, & Jones, 2000). The Theory of Planned Behavior was to be used as a backbone to analyze the results of the study and the survey questions were designed to fit within the three pillars of the theory: (1) a person's attitude towards a behavior, (2) their subjective norm, and (3) their perceived behavioral control govern their intention to express a behavior (Ajzen, 1991). There is no literature on the application of the theory to facilities managers and water or wastewater management. However, the following three studies serve as a guide for this research question. Ishii & Boyer were able to use the Theory of Planned Behavior to quantify the support for urine diversion by University of Florida students (S. K. L. Ishii & Boyer, 2016). In this study, they concluded that three determinants were able to predict a person's acceptance of urine diversion: (1) high frequency of pro-environmental behaviors, (2) perception of a supportive community towards urine diversion, and (3) a belief that votes in favor of urine diversion would influence action by the university (S. K. L. Ishii & Boyer, 2016). Following that study, Simha et al. used the Theory of Planned Behavior to asses support for urine diversion in South India and found that 68% of those surveyed agreed with recycling urine (Simha, Lalander, et al., 2018). By using the theory in the water sector, Lynne, et al. found that when studying water conservation among strawberry farmers, actual control over the system was an important factor to measure, in addition to gauging their perceived control (Lynne, Franklin Casey, Hodges, & Rahmani, 1995). Greaves, et al. also found that the theory could be applied to individuals within an organization to identify how the organization can implement sustainable goals (Greaves, Zibarras, & Stride, 2013). Based on the literature, it is clear that the Theory of Planned Behavior could be applied to facilities managers with a focus on urine diversion systems.

Incremental Steps Towards Radical Change

This dissertation sought to fill gaps of knowledge that would aid in the implementation of urine diversion systems. In the past, urine diversion systems have been implemented as a radical change to the existing system; often executed as complete retrofits with available technologies and current understanding of urine chemistry. A majority of the systems implemented did not fare well, either due to failure of the collection system or a break in the connection between the urine collectors and the end users of the urine-derived products. However, it can be argued that "incremental steps" towards urine diversion can be used to reduce points of failure and ensure the effectiveness of systems in the future. Incremental changes to the technology, as it faces pressures from the surrounding environment, move the technology through each transitional level and towards more and more widespread adoption (Geels, 2002). If the technology cannot adapt to the stressors and pressures, it will fail and will ultimately fall out of existence. Therefore strategic niche management is needed to ensure that novel technologies, i.e. urine diversion, succeed in changing the landscape.

One of the most important aspect of strategic niche management is to allow the technology to shift and evolve based on landscape stressors in an effort to avoid path dependencies that would lead to technological failure (Kemp et al., 1998). Urine diversion systems should be implemented with the assumption that aspects of the system are flexible and able to change "incrementally." Strategic niche management allows for a space for development without the presumption of success at the point of installation. This awareness of creating a safe space allows for technologies to be shielded, nurtured, and empowered (Smith & Raven, 2012). This type of management would allow for the

slow, yet intentional implementation of urine diversion systems. Decision-makers need to understand that some initial pressures may exist, such as an initial dissatisfaction from users. By strategically managing the niche, decision-makers would understand that the system would need to be retrofitted throughout its early stages to allow for robustness as it responds to tensions from actors within and outside the system. This avoids technological lock-in and decreases the existence of path dependencies that are detrimental for the unique nature of each new urine diversion system that is implemented (Smith & Raven, 2012).

Once the management of the technological transition is established, the next step towards implementation that has to be considered is its connection to existing infrastructure. There are two main ways in which urban infrastructure is retrofitted with emerging technologies: (1) the "piecemeal" approach, where projects are installed on a case-by-case basis, or (2) the "systematic" approach, where there is a methodical approach towards installing novel systems (Eames, Dixon, May, & Hunt, 2013). Across the world, urine diversion systems have been implemented as "piecemeal" systems by early adopters. Eames et al. describe the characteristics of these two types of systems but the most unique aspect of systematic systems is their ability to exist in both new construction and retrofits, as opposed to piecemeal systems, which exist mostly in new construction. Currently, urine diversion systems are not easily implemented as retrofits to existing infrastructure and overcoming this disadvantage could have a direct relationship towards more implementations of urine diversion systems.

Strategic niche management can be a way of ensuring that urine diversion systems withstand outside pressures and become a commonplace technology. This would

ultimately become the "incremental steps" needed for "radical change." Urine diversion systems currently sit within a very specialized niche of scientific research and early adopters. Transitioning outside of the niche would mean that all stakeholders within the systems realize the benefits of the system. Applications of urine diversion systems have followed a "piecemeal" approach, which makes it hard to shift the way the public thinks about urine diversion because success is seen with completion of one project. Changing perceptions and attitudes, in combination with more technology development, can aid in moving urine diversion to the "systematic" approach of implementation. And finally, a paradigm shift is needed that creates a society that welcomes new technologies with new habits in order to move our world into a sustainable future (Kuhn, 1962). *New habits* + *new technology* = *prospect for a sustainable planet earth* (*Newton*, 2007).

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APPENDIX A

SUPPORTING INFORMATION FOR CHAPTER 3: REAL-TIME MONITORING AND CONTROL OF UREA HYDROLYSIS IN CYBER-ENABLED NONWATER URINAL SYSTEM

MATERIALS AND METHODS

The hydraulic retention time (HRT) of the system was calculated by the following equation:

$$HRT = \frac{V}{Q}$$
$$V = Volume (mL)$$
$$Q = Flow Rate \left(\frac{mL}{min}\right)$$

The flow rate was calculated by dividing the volume of urine (mL) that enters the urinal testbed in 1 h by 60 min. The total volume of the system was calculated by adding the volume of the urinal trap and the two additional traps. The HRT was found to be approximately 65 min. Table S2 shows the volumes at all locations of the testbed if urinations (V= 237 mL) occur every 10 min, as seen in the mimicking urea hydrolysis experiment. The urinal trap holds 450 mL. Trap 1 and Trap 2 hold 550 mL each. It is assumed that once a trap is filled, the volume that enters the trap equals to the volume that exits the trap. The table shows that urine enters the storage tank after the t = 60 min urination event. This coincides with the 65 min HRT that was calculated based on urination volumes, volume of the system, and time.

RESULTS

Chemical equilibrium software results. In order to verify results found in the calcium hydroxide experiment, Visual Minteq was used to model the chemistry of urine when calcium hydroxide is added. The inputs included all the components of synthetic urine, including the ammonia concentrations found in the grab samples, and the calcium hydroxide addition. pH was kept fixed at pH 12.5. Table S2 below shows the saturation indexes for possible minerals that could precipitate in the system. The minerals in red are oversaturated and the minerals in black are undersaturated. From Table S2, it can be seen that calcium phosphate, magnesium oxide, and magnesium phosphate minerals are oversaturated and could precipitate in the system.

Predictive control logic models. Four lasso regression models were created to control pH of urine in the urinal testbed.(Li, Saetta, Mirchandani, & Boyer, 2019) The models used the most recent 20 pH measurements (variables x_0-x_{19}), the most recent 20 conductivity measurements ($x_{20}-x_{39}$), the time since the last urination event (x_{40}), and the pump time on for the last urination event (x_{41}). The four models are given below with the coefficients in scientific notation:

pH step 1: 6.655-(8.99E-5)x₁-(7.74E-4)x₅-(7.67E-5)x₁₂-(3.60E-3)x₁₃-(4.84E-2)x₁₈+(2 .82E-1)x₁₉+(4.82E-4)x₄₀-(5.95E-4)x₄₁

pH step 2: 6.655-(9.94E-4)x₂-(1.48E-3)x₅-(3.67E-5)x₁₀-(1.08E-3)x₁₁-(1.48E-3)x₁₂+(2 .31E-1)x₁₉+(2.96E-4)x₄₀-(1.10E-3)x₄₁

pH step 3:

 $6.655 - (1.92E - 3) x_2 - (4.17E - 4) x_3 - (1.12E - 3) x_5 - (4.98E - 3) x_{12} - (1.72E - 3) x_{13} + (2.33E - 1) x_{19} + (7.19E - 4) x_{40} - (1.85E - 3) x_{41}$

pH step 4:

 $6.656 - (1.82E - 3) \times_{0} - (9.88E - 4) \times_{3} - (7.62E - 4) \times_{4} - (7.78E - 4) \times_{5} - (5.51E - 3) \times_{12} - (5.57E - 3) \times_{13} + (2.35E - 1) \times_{19} + (9.19E - 4) \times_{40} - (2.65E - 3) \times_{41}$

Sensitivity analysis for first-order rate constant. A sensitivity analysis was conducted to determine the effects of first-order rate constant, k, on the results of the mechanistic model. High and low k values were taken from Yadav, et al and Singh, et al for urea hydrolysis rate experiments in soil.(Singh, Singh, & Singh, 1992; Yadav, Kumar, Singh, & Relan, 1987) Three percent change scenarios were also calculated by changing the experimental k constant used in this study by 10%, 20%, and 50%. Results are shown in Fig. S2. The results show that the rate constant must be determined from experimental data because most rate constants for urea hydrolysis have been found from soil systems. Until a wide variety of urine diversion systems are studied, it is best to determine a case-specific rate constant for urea hydrolysis in the urine diversion system.

Chemical cost. Chemical costs were estimated using Alibaba as a reference for the cost of acetic acid and calcium hydroxide. Alibaba was accessed on October 23, 2018. From Alibaba, it was estimated that a metric ton of acetic acid sold for \$500 and a metric ton for calcium hydroxide sold for \$300. Using the concentrations and doses of the chemical additions used in this study, it was determined that the acetic acid chemical cost was lower than the chemical cost for the calcium hydroxide. That is because the dose of calcium hydroxide was 10 times higher than the dose for acetic acid. With the assumed costs, it was calculated that the acetic acid method used 5400 doses per dollar of chemical while the calcium hydroxide method used 2800 doses per dollar.

REFERENCES

- Li, X., Saetta, D., Mirchandani, P. B., & Boyer, T. H. (2019). Exploration of Multiple Machine Learning Techniques in Time Series pH Prediction in Wastewater Piping System. *In preparation*.
- Singh, B., Singh, B., & Singh, Y. (1992). Urease activity and kinetics of urea hydrolysis in some soils from semiarid regions of Northwestern India. *Arid Land Research* and Management, 6(1), 21-33.
- Yadav, D. S., Kumar, V., Singh, M., & Relan, P. S. (1987). Effects of temperature and moisture on kinetics of urea hydrolysis and nitrification. *Australian Journal of Soil Research*, 25, 185-191.

TABLES

Table 3-S1. Synthetic urine recipe

Concentration		
[mmol/L]		
500		
44		
15		
40		
20		
4		
4		

	Urinal trap, V = 450 mL			Trap 1, V = 550 mL			Trap 2, V = 550 mL			Storage
Time, t	V in	V sum	V out	V in	V sum	V out	V in	V sum	V out	V in
0	237	237	0							
10	237	474	24	24	24	0				
20	237	450	237	237	261	0				
30	237	450	237	237	498	0				
40	237	450	237	237	735	185	185	185	0	
50	237	450	237	237	550	237	237	422	0	
60	237	450	237	237	550	237	237	659	109	109
70	237	450	237	237	550	237	237	550	237	237
80	237	450	237	237	550	237	237	550	237	237
90	237	450	237	237	550	237	237	550	237	237

Table 3-S2. Hydraulic retention time simulation for urination events occurring every 10 min. It is assumed that the volume that enters the trap equals the volume that exits the trap once the trap is filled.

Table 3-S3. Results for saturation indexes for minerals in synthetic urine with calcium hydroxide addition. Minerals in red font are oversaturated and minerals in black text are undersaturated.

	log	Sat.		
Mineral	IAP	Index		
Anhydrite	-7.189	-2.829		
Brucite	21.085	3.985		
Ca3(PO4)2 (am1)	-21.708	3.792		
Ca3(PO4)2 (am2)	-21.708	6.542		
Ca3(PO4)2 (beta)	-21.708	7.212		
Ca4H(PO4)3:3H2O(s)	-42.683	5.267		
CaHPO4(s)	-20.967	-1.692		
CaHPO4:2H2O(s)	-20.973	-1.978		
Epsomite	-6.344	-4.218		
Gypsum	-7.194	-2.584		
Halite	-2.332	-3.882		
Hydroxyapatite	-22.451	21.882		
KCl(s)	-2.707	-3.607		
Lime	20.225	-12.475		
Mg(OH)2 (active)	21.085	2.291		
Mg2(OH)3Cl:4H2O(s)	28.522	2.522		
Mg3(PO4)2(s)	-19.119	4.161		
MgHPO4:3H2O(s)	-20.113	-1.938		
----------------	---------	--------		
Mirabilite	-4.829	-3.715		
Periclase	21.088	-0.497		
Portlandite	20.222	-2.482		
Struvite	-13.16	0.1		
Thenardite	-4.803	-5.124		

Table 3-S4. Mechanistic model parameters used for cases presented in Figure 2-3.

Description	Mimicking urea	Variable urination	Variable urea	
Parameter	hydrolysis	volume	concentration	
Volume, urinal trap, mL	450	450	450	
Volume, trap 1, mL	550	550	550	
Urination volume, mL	237	118.5–474	237	
Urination frequency, min	10	10	10	
Urea concentration, mmol as N	500	500	400–600	
Flow rate, mL/min	711	711	711	
k, 1/s	$8 imes 10^{-6}$	$8 imes 10^{-6}$	$8 imes 10^{-6}$	

Table 3-S5. Random selections for mechanistic model. The program chose a random urination time between 10–40 s and a random urea concentration between 400–600 mmol/L as N. The calculated random urination volume was calculated using a flow rate of 711 mL/min.

Time, min 10 20	Random urination time, s 18 21	Calculated random urination volume, mL 213.3 248.85	Random urea concentration, mmol/L as N 501 593
30	31	367.35	408
40	14	165.9	450
50	31	367.35	405
60	29	343.65	470
70	34	402.9	476
80	10	118.5	547
90	23	272.55	452
100	39	462.15	431
110	36	426.6	521
120	10	118.5	437
130	25	296.25	491
140	38	450.3	435
150	19	225.15	591
160	13	154.05	474
170	34	402.9	579
180	10	118.5	421

190	13	154.05	495
200	13	154.05	531
210	33	391.05	562
220	25	296.25	561
230	22	260.7	470
240	17	201.45	554
250	32	379.2	520



Figure 3-S1. pH and conductivity results for the mimicking urea hydrolysis experiment in trap 2 (a, b) and storage tanks (c, d). Data is shown for each urinal. The solid lines correspond to the values at t = 0 of the synthetic urine. Data points were taken every 1 min.



Figure 3-S2. pH and conductivity results for the inhibiting urea hydrolysis with acetic acid experiment in trap 2 (a, b) and storage tanks (c, d). Data is shown for each urinal. The solid lines correspond to the values at t = 0 of the synthetic urine. Data points were taken every 1 min. Data for urinal 1 not shown due to technical difficulties with the sensor controller.



Figure 3-S3. pH and conductivity results for the inhibiting urea hydrolysis with calcium hydroxide experiment in trap 2 (a, b) and storage tanks (c, d). Data is shown for each urinal. The solid lines correspond to the values at t = 0 of the synthetic urine. Data points were taken every 1 min.





Figure 3-S4. Results for the mimicking and inhibiting experiments with no treatment, acetic acid addition, and calcium hydroxide addition. Concentrations for ammonia, phosphate, calcium, magnesium, sodium, and potassium in grab samples from the fresh urine at t = 0 (FU), storage tank 1 at t = end (S1), storage tank at t = end (S2), storage tank at t = end (S3) and the fresh urine at t = end (FU END) are shown. Ammonia and phosphate concentrations are the average concentrations found when the same sample was run two times on the FIA. Error bars correspond to one standard deviation.



Figure 3-S5. Mechanistic model assumptions for the steps in calculation. New urine displaces old urine in plug flow manner. It instantaneously mixes to form a new concentration. Reaction occurs for 10 min. Urine at t_{final} corresponds to the urine that has mixed and reacted with the old urine. Progression begins again with every new urination.



Figure 3-S6. Results of sensitivity analysis of the first-order rate constant, k, on the final ammonia concentration in the urinal trap. The centerline corresponds to the concentration of ammonia calculated by using the experimental k-constant used in the mechanistic models ($k = 8 \times 10^{-6} \text{ s}^{-1}$). The percent change scenarios correspond to the respective percent change on the experimental k-constant.



Sensitivity Analysis: First-order rate constant



Figure 3-S7. Results for the reactive and predictive control logic experiments at low and high frequencies. Normalized concentrations for ammonia, phosphate, calcium, magnesium, sodium, and potassium in grab samples taken every 30 min. Ammonia concentrations were normalized by the total N in synthetic urine. All other concentrations were normalized by the concentration in the synthetic urine at t = 0. Each bar is the average concentration from the three urinals or storage tanks. Trap 1 (orange bars), trap 2 (dashed bars), and the storage tank (white bars) are shown with error bars corresponding to one standard deviation.



Figure 3-S8. Cumulative volume (L) versus normalized time for the three experiments using reactive or predictive control logic at low and high frequencies. The high use, reactive control logic experiment is shown as the purple circles, the low use, reactive control logic experiment is shown as the green triangles, and the low use, predictive control logic experiment is shown as yellow squares.





Figure 3-S9. Results for the reactive and predictive control logic experiments at low and high frequencies. Concentrations for ammonia, phosphate, calcium, magnesium, sodium, and potassium in grab samples from the fresh urine at t = 0 (FU), storage tank 1 at t = end (S1), storage tank at t = end (S2), storage tank at t = end (S3) and the fresh urine at t = end (FU END) are shown. Ammonia and phosphate concentrations are the average concentrations found when the same sample was run two times on the FIA. Error bars correspond to one standard deviation.



Figure 3-S10. Front of the urinal testbed. Pumps for urine, urease, and acid are on the right. Two pumps for grab samples are on the left.



Figure 3-S11. Back of urinal testbed. Two-inch cast iron pipe is used to convey the urine from the urinals to the storage tanks. Two P-traps were used to hold pH and conductivity sensors in urine solution.

APPENDIX B

SUPPORTING INFORMATION FOR CHAPTER 4: IMPACT OF ACETIC ACID ADDITION ON NITROGEN SPECIATION AND BACTERIAL COMMUNITIES DURING URINE COLLECTION AND STORAGE

FIGURES



Figure 4-S1 – The urinal-on-wheels inside the restroom stall where it was placed during urine collection.



Figure 4-S2 – Inside the urinal-on-wheels. The urine tank was outfitted with pH and temperature sensors. The motion sensors and the button were mounted to face the outside of the box. The sensors were battery powered and controlled with a Raspberry Pi microprocessor. The plastic bottle was filled with activated carbon to allow for airflow as urine entered the tank.



Figure 4-S3 – The glass urinal prior to urine collection. Nonwater urinal sealing liquid was placed in the "trap" and can be seen as the green fluid in the picture.



Figure 4-S4 - A heatmap of average Wifi logins per hour and day of the way for the building floor on which the restroom studied is found.



Figure 4-S5 – The temperature (°C) of urine collected with the urinal-on-wheels for Runs 1-8. Data points were taken every 5 min.



Figure 4-S6 – Phosphate concentrations for daily urine grab samples during urine collection. Error bars show the standard deviation for duplicate sample analysis.



Figure 4-S7 – Total organic carbon concentrations for daily urine grab samples during urine collection. Error bars show the standard deviation for duplicate sample analysis.



Figure 4-S8 – pH and conductivity measurements of urine samples collected from the urine "tank" in the glass urinal experiments. "Baseline" experiments monitored urea hydrolysis without acid addition. "Acid" denoted runs studying the impact of acid addition after each urination event. Three runs of each experiment were conducted. The samples used for bacterial analysis were Baseline 2 and Acid 3.



Figure 4-S9 – Total nitrogen, ammonia, phosphate, and total organic carbon concentrations for urine samples taken at the end of each glass urinal run. Error bars for the standard deviation in duplicate analysis is shown on the bars.

APPENDIX C

SUPPORTING INFORMATION FOR CHAPTER 5: DATA MINING METHODS PREDICT CHLORINE RESIDUALS IN PREMISE PLUMBING USING LOW-COST SENSORS

TABLES & FIGURES

Model						
.,	Floor	No. of	Tree	Shrinkage	Min. no. observations at	Subsampling
#		trees	depth		terminal nodes	
10	2	45	3	0.1	5	0.65
11	2	258	5	0.3	7	0.85
12	2	11	6	0.4	15	0.60
13	3	21	1	0.2	7	0.65
14	3	571	5	0.1	5	0.65
15	3	9	5	0.4	15	0.65
16	7	6	3	0.3	3	0.50
17	7	6	7	0.3	8	0.75
18	7	7	5	0.3	5	0.80

Table 5-S1– Summary table of final gradient boosting machine model parameters. Final GBM model parameters

	Boxplot Stats	рН	Conductivity, uS/cm	Temperature, °C	Free chlorine, mg/L	DO, mg/L	ORP, mV	Wifi Logins
	Min.	7.34	287	20.3	0.00	8.20	527	0
2nd Elear	1st. Quadrant	7.46	611	21.0	0.33	8.84	724	1
(979	Median	7.64	908	21.7	0.46	9.08	733	6
obs.)	Mean	7.60	850	21.5	0.43	9.25	724	17.1
	3rd. Quadrant	7.73	1058	22.0	0.52	9.73	740	22
	Max	7.82	1245	22.5	0.81	10.9	757	131
			r					
	Min.	7.53	350	20.7	0.00	7.21	237	0
ard Eleor	1st. Quadrant	7.78	741	23.3	0.00	9.24	379	0
(665	Median	7.82	817	24.3	0.08	9.62	448	2
obs.)	Mean	7.81	860	24.2	0.12	9.53	474	11.2
005.)	3rd. Quadrant	7.84	992	25.0	0.18	9.99	555	13
	Max	7.96	1729	28.6	0.62	10.5	759	139
	Min.	7.57	555	22.6	0.00		0.650	0
7th Floor	1st. Quadrant	7.75	724	24.6	0.00		92.6	1
(1099	Median	7.80	972	25.2	0.00		279	4
obs.)	Mean	7.79	942	25.3	0.01		268	9.3
	3rd. Quadrant	7.83	1123	25.9	0.00		396	11
	Max	7.98	1572	27.7	0.07		649	106

Table 5-S2 – Preprocessed water quality and Wifi login data for the 2nd floor, 3rd floor, and 7th floor prior to normalization.

Table 5-S3 – Welch's t-test p-values for each pair of floors for the water quality parameters that were measured and the Wifi login data was collected. P-values < 0.05 indicate significant difference. Bold results indicate samples with no significant difference.

Welch's t-test p-values for water quality and Wifi logins				
Temp. floor pairs	Temp. floor pairs p-value		p-value	
2 nd floor & 3 rd floor	< 2.2E-16	2 nd floor & 3 rd floor	< 2.2E-16	
2 nd floor & 7 th floor	< 2.2E-16	2 nd floor & 7 th floor	< 2.2E-16	
3 rd floor & 7 th floor	< 2.2E-16	3 rd floor & 7 th floor	< 2.2E-16	
pH floor pairs	p-value	DO floor pairs	p-value	
2 nd floor & 3 rd floor	< 2.2E-16	2 nd floor & 3 rd floor	< 2.2E-16	
2 nd floor & 7 th floor	< 2.2E-16	2 nd floor & 7 th floor	-	
3 rd floor & 7 th floor	3.19E-11	3 rd floor & 7 th floor	-	
Cond. floor pairs	p-value	Wifi floor pairs	p-value	
2 nd floor & 3 rd floor	0.3819	2 nd floor & 3 rd floor	1.15E-07	
2 nd floor & 7 th floor	< 2.2E-16	2 nd floor & 7 th floor	< 2.2E-16	
3 rd floor & 7 th floor	1.70E-14	3 rd floor & 7 th floor	0.02865	
ORP floor pairs	p-value			
2 nd floor & 3 rd floor	< 2.2E-16			
2 nd floor & 7 th floor	< 2.2E-16			
3 rd floor & 7 th floor	< 2.2E-16			

	Adjusted R ²						
	Wifi logins	Wifi logins	Wifi logins	Wifi logins	Wifi logins	Wifi logins	
Floor	& pH	& ORP	& Cond	& Temp	& Chlorine	& DO	
2nd floor	0.01	4.4E-03	0.12	0.13	2.5E-04	0.11	
3rd floor	0.03	0.07	0.07	0.18	0.44	2.5E-03	
7th floor	3.7E-03	0.02	0.02	-2.8E-04	0.07	_	

Table 5-S4 – Summary table of adjusted R^2 values for the regression lines shown in Figure 5-4.

Figure 5-S1 – Floor plans for each floor in the study building. The water quality sensor platform was installed in the break room on the 2^{nd} , 3^{rd} , and 7^{th} floor.





7th floor



2nd Floor Correlation Coefficients

Figure 5-S2 - Plots for the 2nd floor showing the normalized raw data points, the variable distribution on the diagonal, and the Pearson correlation coefficient for the pairs in the raw data.



3rd Floor Correlation Coefficients

Figure 5-S3 - Plots for the 3^{rd} floor showing the normalized raw data points, the variable distribution on the diagonal, and the Pearson correlation coefficient for the pairs in the raw data.



Figure 5-S4 – Plots for the 7th floor showing the normalized raw data points, the variable distribution on the diagonal, and the Pearson correlation coefficient for the pairs in the raw data.



Figure 5-S5 – Histogram of the DO concentration measurements for the 7th floor.



Figure 5-S6 – Linear regression modeling data versus measured data for models using Wifi logins only (models 1, 4, and 7). The 1:1 line is displaying on each graph. Chlorine (mg/L) = $0.52 + (0.05 \times \text{Wifi})$ Chlorine (mg/L) = $0.11 + (1.08 \times \text{Wifi})$ Chlorine (mg/L) = $0.05 + (0.41 \times \text{Wifi})$


Figure 5-S7 – Linear regression modeling data versus measured data for models using all parameters (models 3, 6, and 9). The 1:1 line is displaying on each graph. Chlorine (mg/L) = $0.15 + (0.09 \times \text{Wifi}) + (0.36 \times \text{ORP}) + (0.17 \times \text{pH}) + (0.25 \times \text{Cond}) + (-0.28 \times \text{Temp}) + (-0.09 \times \text{DO})$ Chlorine (mg/L) = $-0.19 + (0.82 \times \text{Wifi}) + (0.32 \times \text{ORP}) + (0.21 \times \text{pH}) + (-0.15 \times \text{Cond}) + (-0.25 \times \text{Temp}) + (0.29 \times \text{DO})$ Chlorine (mg/L) = $0.23 + (0.39 \times \text{Wifi}) + (0.14 \times \text{ORP}) + (-0.29 \times \text{pH}) + (0.003 \times \text{Cond})$

Chlorine (mg/L) = $0.23 + (0.39 \times \text{Wifi}) + (0.14 \times \text{ORP}) + (-0.29 \times \text{pH}) + (0.003 \times \text{Cond}) + (-0.14 \times \text{Temp})$

APPENDIX D

INSTITUTIONAL REVIEW BOARD (IRB) APPROVAL FOR HUMAN SUBJECT TESTING



APPROVAL: EXPEDITED REVIEW

Treavor Boyer Sustainable Engineering and the Built Environment, School of (SEBE)

thboyer@asu.edu

Dear Treavor Boyer:

On 12/6/2016 the ASU IRB reviewed the following protocol:

Type of Review:	Initial Study
Title:	Nutrient Removal and Recovery from Source Separated Urine
Investigator:	Treavor Boyer
IRB ID:	STUDY00005328
Category of review:	(3) Noninvasive biological specimens
Funding:	Name: Arizona State University (ASU)
Grant Title:	
Grant ID:	
Documents Reviewed:	 IRB Recruitment Email and Flyer.pdf, Category: Recruitment Materials; IRB Application Revised.docx, Category: IRB Protocol; Treavor Boyer CITI Training 10-30-15.pdf, Category: Non-ASU human subjects training (if taken within last 3 years to grandfather in); Daniella Saetta CITI Training 4-18-16.pdf, Category: Non-ASU human subjects training (if taken within last 3 years to grandfather in); Daniella Saetta CITI Training 4-18-16.pdf, Category: Non-ASU human subjects training (if taken within last 3 years to grandfather in); Showing Changes, Category: Consent Form; Final Clean Version, Category: Consent Form; Point by Point Response Letter to Changes, Category: Other (to reflect anything not captured above);

The IRB approved the protocol from 12/6/2016 to 12/5/2017 inclusive. Three weeks before 12/5/2017 you are to submit a completed Continuing Review application and required attachments to request continuing approval or closure.

If continuing review approval is not granted before the expiration date of 12/5/2017 approval of this protocol expires on that date. When consent is appropriate, you must use final, watermarked versions available under the "Documents" tab in ERA-IRB.

In conducting this protocol you are required to follow the requirements listed in the INVESTIGATOR MANUAL (HRP-103).

Sincerely,

IRB Administrator

cc: Hannah Ray Neha Jagtap Hannah Ray Avni Solanki Daniella Saetta



APPROVAL: MODIFICATION

<u>Treavor Boyer</u> <u>SEBE: Sustainable Engineering and the Built Environment, School of</u>

thboyer@asu.edu

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Dear <u>Treavor Boyer</u>:

On 2/14/2020 the ASU IRB reviewed the following protocol:

Type of Review:	Continuing Review
Title:	Nutrient Removal and Recovery from Source
	Separated Urine
Investigator:	Treavor Boyer
IRB ID:	STUDY00005328
Funding:	Name: Arizona State University (ASU)
Grant Title:	None
Grant ID:	None
Documents Reviewed:	• Final Clean Version, Category: Consent Form;
	Showing Changes, Category: Consent Form;

The IRB approved the modification.

When consent is appropriate, you must use final, watermarked versions available under the "Documents" tab in ERA-IRB.

In conducting this protocol you are required to follow the requirements listed in the INVESTIGATOR MANUAL (HRP-103).

Sincerely,

IRB Administrator

cc: Daniella Saetta Uranus Richard Rebecca Dietz Urusha Regmi Angela Egan Neha Jagtap Hannah Ray Lerys Del Moral Michael Edgar Daniella Saetta