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MICROCOSM ASSESSMENT OF NATURAL PROCESSES AFFECTING CHEMICALS OF EMERGING CONCERN IN SECONDARY EFFLUENT

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SCHOOL OF CIVIL ENGINEERING AND ENVIRONMENTAL SCIENCE

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Thanks mom and dad. We did it!

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Abstract

The fight for new water sources is over, reclaimed water is next. Currently, the City of Norman supports its water needs via Lake Thunderbird (LT) and the Central Oklahoma Aquifer System (COAS). In times of peak demand, the City of Norman can purchase treated drinking water from Oklahoma City, but state-wide drought threatens the longevity of this water source. In response, the City of Norman contracted an engineering firm to draft a strategic water supply plan. Several portfolios were produced, but the most promising one called for the augmentation of the potable water supply through indirect potable water reuse. A portion of the effluent from the Norman Water Reclamation Facility would be discharged into an environmental buffer, Dave Blue Creek. Currently, the Norman Water Reclamation Facility is not designed for the removal of chemicals of emerging concern (CEC) which are detected in effluent at trace amounts (ng/L parts per trillion or ppt).

Microcosm studies were setup to simulate the behavior of 98 CEC when incubated with Dave Blue Creek sediment and photosynthetically active radiation lights. Erlenmeyer flasks were filled with treated effluent and sediment fractions less than 0.25 mm with a 2:1 ratio w/v. The flasks were incubated for 15 days on an orbital shaker at 125 rpm. Sorption (SED) and photodegradation (PAR + SED and PAR + Effluent) microcosms were run separately.

Photodegradation appeared to be the most effective pathway for CEC detection reduction. When evaluated by class (e.g., EDCs, PPCPs, stimulants,

preservatives, sweeteners, pesticides, and flame retardants) the effects of sorption and photodegradation varied. Sorption appeared to be an important pathway for decreasing the number of pesticide detection. The effects of photodegradation seemed most effective at reducing PPCP detections. EDC, flame retardant, and preservative detects were susceptible to the synergistic effects of photodegradation and sorption, and attenuation of artificial sweeteners and stimulants were negligible for both mechanisms.

Chapter 1. Introduction

The City of Norman is on the brink of a public water supply crisis. Currently, residents are supplied water via Lake Thunderbird (LT) and groundwater wells from the Central Oklahoma Aquifer System (COAS) (Carollo, 2014). Norman is experiencing a deficit meaning that the demand for water exceeds the municipality's ability to provide for its residents. In the past, bridging this gap meant purchasing treated water from Oklahoma City. However, even with conservation methods, reliance on Oklahoma City's pipeline will not be able to sustain the projected increases in population and water demand of both cities. Further, groundwater availability in the COAS is expected to be at half its capacity within the next 35 years (Mashburn et al., 2013). In addition, climatic change, and the timing, magnitude, and location of precipitation events could affect water availability and demand patterns in Oklahoma overall (Carollo, 2015).

In response, the City of Norman contracted an engineering firm to draft the Norman Utilities Authority 2060 Strategic Water Supply Plan (SWSP) (Carollo, 2014). Through this effort, the engineering firm evaluated several alternatives, called portfolios, to address future water needs of the city. Out of the 15 portfolios drafted, two portfolios were identified for final consideration: portfolio 13 (P13) and portfolio 14 (P14). The first, P13, called for a joint venture of Norman and Oklahoma City to pipe raw water from Southeast Oklahoma. P14 called for augmentation of Lake Thunderbird via highly treated wastewater effluent and

additional groundwater wells. A portion of the effluent from the Norman Water Reclamation Facility (NWRF) would be diverted into Dave Blue Creek (DBC) and flow by gravity into LT, and eventually be treated at the Norman Water Treatment Plant (Carollo, 2014). Although both were considered viable options, P13 had higher upfront costs, reliance on another municipality, as well outstanding water rights disputes making it the less desirable option (Carollo, 2014; Layden, 2014). Thus, P14 was deemed the top ranked portfolio. Nevertheless, reclaimed water coming into direct contact with consumers is a major concern for the public (Buyukkamaci and Alkan, 2013). As Dr. Lucas van Vuuren, scientist at the National Institute of Water Research in South Africa, said, "Drinking water should not be judged by its history, but by its quality" (Jiménez and Asano, 2008).

Water reuse is often associated with the "toilet to tap" concept, giving this practice a controversial reputation. Social acceptance aside, more understanding on CEC is needed. CEC are a wide array of synthetic or natural unregulated chemicals frequently detected in surface waters in trace amounts (Cullin, 2014) that are not removed by conventional wastewater treatment systems (AWWA, 2007; Tchobanoglous, 2015; USGS, 2017). This research sought to model the fate of highly treated effluent, without polishing treatment, in a natural environmental buffer. Ninety-eight CEC were analyzed over a 15-day period to identify the effects of sorption and photodegradation. The data represent a "worst case" scenario of water reuse implementation in Norman, Oklahoma. Worst case stems from two things. First, limited pathways were provided for CEC mitigation within the

microcosms. There are multiple forms of attenuation, but sorption and photodegradation were the focus of this research. Second, the effluent was collected as is, without any polishing treatments (e.g., advanced oxidative processes, or reverse osmosis) that would be implemented prior to enacting P14. Thus, the quantity of CEC tested in the microcosms and concentrations of compounds selected for analysis would most probably be higher than effluent from a system designed for greater percent removal of CEC.

Chapter 2. Literature Review and Background

2.1 Water Reuse

Water reuse has the potential to reduce demand on traditional water supplies by increasing the total available water resources on the planet (NRC, 2012; Garcia-Cuerva et al., 2016). The agricultural industry has reused water for irrigation purposes (Brahim-Neji et al., 2014). Freshwater extracted from produced water in the oil and gas industry, has mitigated competition with agricultural, municipal and industrial consumers (Mason, 2016). Even though non-potable municipal effluent has been used for irrigation and industrial purposes for many decades, people have been conditioned to separate drinking water and municipal effluent because of social perception of reclaimed water (Hawker et al., 2011). Albeit, incidental potable reuse (PR) has been practiced in the United States for a long time (NRC, 2012).

Reuse intended for public water supply (PWS) has two main categories, direct and indirect. Direct potable reuse (DPR) is the introduction of treated reclaimed water into the PWS. When the citizens of Wichita Falls, Texas needed emergency water, plant operators took a portion of chlorinated effluent and treated it with microfiltration, reverse osmosis, and UV disinfection before further treatment at a drinking water treatment facility (Tchobanoglous et al., 2015). This use of DPR tends to be less favorable when indirect potable reuse (IPR) is an option to increase the PWS due to the “toilet to tap” concept (Hawker et al., 2011).

IPR is the introduction of treated effluent into a natural buffer before being introduced to the PWS. For decades, California has mitigated saltwater intrusion in aquifers by recharging groundwater with effluent. Since 1978, Fairfax County, Virginia has augmented surface waters with treated effluent (Tchobanoglous et al., 2015). Arizona, Colorado, Florida, and Nevada have also implemented IPR systems (Jones, 2016; Metcalf and Eddy, 2007; NRC, 2012). Globally, Europe, Australia and Singapore have also implemented IPR for PWS augmentation (Jones, 2016). Generally, IPR is more popular than DPR due to the associated repugnance of water reclamation facilities. Environmental buffers connect the water to its history, which tends to ease public perception (NRC, 2012). The natural environment also helps increase the amount of time between PWS inclusion, dilute treated effluent, and naturally attenuate contaminants (NRC, 2012). Qualities such as these are important to reuse projects like P14.

2.2 Chemicals of Emerging Concern Background

CEC analytes have been documented in wastewater since the 1960s, but only received attention when their presence in effluent at nanogram per liter (ng/L) or part per trillion (ppt) concentrations was reportedly affecting in the reproduction of aquatic biota (AWWA, 2007). More specifically, CEC in surface waters have caused the feminization of male fish, breakage of fish, bird, and turtle eggs, as well as reproductive issues and immune system changes in marine biota (Espulgas, 2007; AWWA, 2007). Overall, CEC have the capacity to affect population growth of aquatic animals and disrupt the harmony of entire ecosystems.

CEC have likely been in the environment for a long time, but now that analytical techniques have improved to detect and quantify trace CEC amounts they are more widely studied (AWWA, 2007). These chemicals may be future candidates for regulation depending on ecotoxicity, potential threats to human health, and frequency of occurrence (Tchobanoglous, 2015). The uncertainty surrounding the human health-based risks associated with CEC do not qualify them for monitoring under the Safe Drinking Water Act (EPA, 2015), but future regulatory standards may require tertiary treatment of CEC in recycled water (AWWA, 2007). Currently, influent at the NWRF receives primary and secondary treatment. These conventional wastewater treatment practices are not designed to remove CEC, which are subsequently discharged into the environment, which adds to public speculation and doubt regarding water reuse.

2.2.1 Characterization of CEC

The pharmaceutical industry alone obtained approval from the Food and Drug Administration (FDA) for 1,453 drugs from 1827–2013 (Gaffney, 2014), and pharmaceuticals only account for a portion of the CEC present in the environment today. Moreover, there are “over 84,000 chemicals, as inventoried by the EPA under the Toxic Substances Control Act (TCSA)” (Jones, 2016). Since they are so numerous, CEC are often lumped into categories (NRC, 2012; Laubacher, 2016; Jones, 2016): endocrine disrupting chemicals (EDCs), pharmaceuticals and personal care products (PPCPs), stimulants, preservatives, artificial sweeteners, pesticides, and flame retardants. Further, characterization can be broken down by use. The

categories help aid in the general understanding of how certain CEC behave in the environment.

2.2.2 Occurrence in the Norman Water Reclamation Facility

Laubacher (2016) performed a study geared towards monitoring and evaluating CEC at the NWRF through conventional activated sludge (CAS) treatment. The study contracted Eurofins Eaton Analytical (EEA) to monitor 98 CEC, but only 39% were indicated above the minimum reporting limit (MRL). Although most of the detected CEC did exhibit considerable reductions in concentrations, there was still detection of trace CEC concentrations in the effluent.

Prior to Laubacher's 2016 study, City of Norman and EEA joined forces and conducted an IPR study to assess the potential impacts of Lake Thunderbird augmentation with treated effluent (Jones, 2016). City officials and EEA came up with the "Norman 96" to conduct the study. This was a comprehensive list of CEC analytes based on studies conducted by the Water Reuse Association (WRA), National Water Research Institute (NWRI), Environmental Protection Agency (EPA), US Geological Survey (USGS), and the US Bureau of Reclamation (BOR) (Jones, 2016). Out of the 96 CEC, 41 were detected in NWRF effluent. In 2016, there were 38 cumulative CEC detections above the MRL at the NWRF, after effluent from each stage in the treatment process was analyzed (Laubacher, 2016). In both cases, a substantial number of CEC were detected at ng/L or ppt concentrations. The Norman 96 became the staple of CEC testing at EEA.

2.3 CEC Regulations

In some places, wastewater was considered a liability. Treatment meant doing the bare minimum to meet discharge standards. This liability, however, is quickly becoming an asset in arid climates, and more thought is being placed on the water rights of people downstream (NRC, 2012). States like California, Nevada, Oregon, Utah, and Washington have enacted laws to address the right to reuse water. In Utah, wastewater cannot be reused by the facility unless specified in a permit; while in Colorado, plant owners are permitted to use treated wastewater within the municipality (NRC, 2012). Although, effluent utilization is state-dependent, states seem to agree that the water quality and quantity of discharge should not impact downstream users. The following sections will review current Federal and Oklahoma state regulations with regards to water reuse.

2.3.1 Federal Regulations

There were no federal regulations governing water reuse as of 2012 (NRC, 2012). The Clean Water Act (CWA) and Safe Water Drinking Act (SWDA) offer protection from pollution and general safety of the public water supply, but do not offer much oversight for reclaimed water. With states considering potable and non-potable augmentation, the EPA adopted "Guidelines for Water Reuse" (NRC, 2012). The proposed guidelines "recommend that PR projects meet drinking water standards and monitor for hazardous compounds (or classes of compounds) not included in the drinking water standards" (NRC, 2012).

Presently, there are no health standards attached to CEC under the SWDA, even though their presence in surface waters has been prevalent for decades. For unmonitored compounds, the EPA uses the Unregulated Contaminant Monitoring Rule (UCMR). Each cycle of contaminants considered for the UCMR is based upon review of the Contaminant Candidate List (CCL) (EPA, 2016a). CCL 1 was developed in 1998, and contained 50 chemicals (EPA, 2016b). Upon review, contaminants remaining on the previous CCL were carried over to the subsequent one. In 2016, the Final CCL 4 was announced, nearly three decades after the first CCL was produced (EPA, 2016b).

2.3.2 Oklahoma Regulations

Water reuse has been a promoted practice in Oklahoma for agriculture and other non-potable municipal uses since the 1970s (ODEQ, 2014a). Anticipating that the state would experience widespread drought, the Oklahoma Department of Environmental Quality (ODEQ), began developing Water Reuse Standards. In the summer of 2012, the ODEQ came out with the Operation and Maintenance of Water Reuse Systems (OAC 252:627) and Water Construction Standards (OAC 252:656-27) (ODEQ, 2014a).

A cross section of industry stakeholders with experience developing reuse regulations were appointed to develop a paper summarizing the status of DPR and IPR to expedite water reuse projects (ODEQ, 2014b). There are three sub categories

used to address PR: IPR surface water, IPR groundwater, and DPR. The document (ODEQ, 2014b) did the following:

1. Provided historical and ongoing research related to PR;
2. Provided information on current state and national efforts to develop regulations and guidelines for IRP and DPR;
3. Identified challenges and questions that need to be addressed related to implementation of PR in Oklahoma; and
4. Developed recommendations for a process and revised timeline for establishing IPR and DPR regulations in Oklahoma.

The paper described surface water IPR as the use of reclaimed water being intentionally discharged into a lake, river, or other water supply (ODEQ, 2014b). The committee was encouraged to focus on the development of Category 1a (Surface Water IPR), since projects of this nature were in higher demand. This meant creating a working definition for IPR, determining if additional treatment was required, amending current water quality standards, and developing guidelines to demonstrate compliance for IPR discharges. Similar considerations were taken for Groundwater IPR (Category 1b). For DPR (Category 1c) recommendations would be made out on a case by case basis, as in other states, until specific guidance is outlined (ODEQ, 2014b). The formal adoption of IPR Regulations was expected during the latter half of 2017, but as of now is still underway.

2.4 Natural Attenuation of CEC

Hawker et al. (2011), assessed the natural attenuation of organic contaminants in Lake Wivenhoe, in South-East Queensland, Australia, using a "Level III fugacity based evaluative fate model". Lake Wivenhoe is a water body used for potable drinking water, like Lake Thunderbird. The region supported by Lake Wivenhoe is experiencing a population increase, placing stress on the water body, as are increased periods of drought. The municipality decided to augment Lake Wivenhoe using IPR to alleviate water scarcity in the region. Aside from dual membrane filtration, and ozonation, it was noted that environmental buffers are the best line of defense for IPR (Hawker et al., 2011). These natural systems are expected to aid in the attenuation of contaminants, but the extent of this degradation was unknown and required further investigation, preferably in situ (Hawker et al., 2011). The study utilized physiochemical parameters of the organic pollutants to determine the fate and transport of "disinfection-by-products, pesticides, PPCPs, xenoestrogens, and industrial chemicals" (e.g., biodegradation, dilution, adsorption, photo transformation), and the main phases considered were water and sediment. The model assumed steady-state conditions, and found attenuation to be linked to volatilization, sorption, and degradation for the detected organic pollutants involved (Hawker et al., 2011). For a good portion of the CEC analyzed, significant fractions were in the sediment (Hawker et al., 2011). Out of 52 PPCPs analyzed, less than 6% were detected, and less than half of the pesticides studied were detected. The organic pollutants across the board experienced a 30-fold reduction, from their initially low

(below health guidelines) concentrations. Hawker et al. (2011) also mentioned that attenuation in a real-world environment would be even higher, but further study would need to be conducted in situ.

Similarly, Yu and Chu (2009) organized a study on the West Prong Little Pigeon (WPLP) River, in Tennessee. They analyzed ibuprofen, caffeine, triclosan (TCS), bisphenol-A, and five other compounds to complete this assessment (Yu and Chu, 2009). The section of the river studied was just outside of a park boundary with two discharge points from wastewater treatment plants (WWTP)that were 8 km apart. Using the treatment capacity of the first WWTP, and the average flowrate of the WPLP River, the authors expected a 50-fold dilution of the compounds in the river. Yu and Chu (2009) also observed that effluent from WWTP1 experienced minimal mixing with the river water because it was discharged close to the riverbank as opposed to effluent from WWTP2 that was discharged closer to the center of the river. Out of the nine PPCPs analyzed, only ibuprofen and TCS were present in the river (Yu and Chu, 2009). The study also illustrated that as the effluent flowed further away from the outfall, estrogenicity decreased (Yu and Chu, 2009). These studies showed that natural attenuation of CEC is possible in water systems. Comparable results were found by the National Research Council (2012). They performed a comprehensive study on water reuse and found natural buffers to not only be successful, but also useful in gaining public acceptance.

Aside from degradation and sorption, photolysis also aids in the attenuation of CEC in the natural environment. Kim and Tanaka (2009) reviewed other works and found the effects of ultraviolet (UV) treatment on CEC and reported the studies to be mildly successful. However, the studies analyzed very few compounds to determine the effectiveness of using UV processes to treat for CEC (Kim and Tanaka, 2009). In their experiment, Kim and Tanaka (2009) examined the treatment of two UV lamps on 30 CEC commonly found in surface waters. All CEC concentrations were quantified using liquid chromatography-tandem mass spectrometry (LC/MS/MS) (Kim and Tanaka, 2009). Five CEC were degraded more than 90% under UV lamp treatment (Kim and Tanaka, 2009). This suggested that some CEC are more susceptible to photodegradation than others. Kim et al. (2009) treated the same 30 CEC with UV/H_2O_2 treatment in a follow-up study. The CEC were irradiated under UV light for 30 minutes with a dose of 691 mJ/cm^2, and 8.2 mg/L of H_2O_2. In these experiments, all but seven PPCPs were degraded by more than 90%. UV/H_2O_2 treatment will not be analyzed in this research, but this is relevant because increasing the number of hydroxyl radicals improves the degradation of PPCPs under UV treatment.

2.5 Ecological Studies of CEC

Although there have been numerous case studies showing that natural attenuation of PPCPs is possible, all ecosystems are not created equal. Therefore, all sites considered for environmental buffers need to be tested for their capacity to degrade CEC (NRC, 2012). Yu et al. (2013) examined the degradation and sorption

of five CEC commonly studied in the environment (bisphenol A, carbamazepine, gemfibrozil, octylphenol, and triclosan). Sorption was modeled by the Freundlich equation using first-order decay. Samples contained varying contents of silt, sand, clay, and organic matter. After running several batch tests, sorption coefficients of carbamazepine (CBZ) and gemfibrozil (GFB) were found to be very low (Yu et al., 2013). With respect to degradation, CBZ was the most persistent in the study, exhibiting a half-life ranging from 28.0–39.1 days, whereas the other contaminants displayed a maximum half-life of 18.0 days (Yu et al., 2013). Sterilized samples from the degradation batch tests increased the CEC persistence in the environment, showing that microorganisms assist in the degradation of CEC in sediment samples (Yu et al., 2013). Yu et al. (2013) calculated the "mean value of single-point distribution coefficient" (K_d) to compare sorption affinities of the compounds. Using the fraction of organic carbon, they calculated normalized distribution coefficients. The sorption of TCS was highest in the sediment containing the greatest fraction of organic carbon.

Similar findings were concluded by Zhang et al. (2013), a group of researchers studying chloramphenicol, caffeine, tinidazole, and metronidazole in the environment. Batch experiments were conducted with soil and sediment to represent a variety of physiochemical processes. The silt-loam soil had the highest concentration of organic carbon and had the highest adsorption capacity. Low to moderate persistence was observed, as well as first-order exponential decay, with a maximum half-life of 10.21 days, and microbial populations were shown to aid in the

degradation of CEC (Zhang et al., 2013). Silty-loam soils in both studies with higher organic carbon content had higher sorption coefficients. Lastly, both studies presented pharmaceuticals that would persist in the environment.

2.6 Public Acceptance of CEC

Environmental buffers are crucial to the success of IPR projects. Although the application of reclaimed water increases PWS, it is not enough to bolster public acceptance of drinking "used" water, environmental buffers do. Public acceptance hinges on the perception that the water is "natural", and when it has passed through an environmental buffer (e.g., natural water body) the consumer confidence is greater (NRC, 2012). The greatest causes of concern for water reuse are potential "health risks associated with recycled water" (Buyukkamaci and Alkan, 2013). When recycled water is supposed to come in "direct contact" with humans it results in opposition (Buyukkamaci and Alkan, 2013; Hartley et al., 2006; Marks, 2006; Stenekes et al., 2006). In a survey analyzed by Buyukkamaci and Alkan (2013), most respondents were fine with water reuse for the purposes of flushing, construction, and cleaning roads. When it came to "direct contact", as in use of reclaimed water for preparing canned food or drinking purposes, there was a great lack of public support (Buyukkamaci and Alkan, 2013). IPR calls for ingestion of some reused water mixed with the potable water supply, therefore, keeping the public involved is important for a successful reuse project. Buyukkamaci and Alkan (2013), found the media to be the preferred choice of channeling information to the potential consumers of reuse water.

2.7 Hypotheses and Objective

In 2015, the Oklahoma Water Survey held a series of brief Public Forums on Water Reuse, at the National Weather Center near the University of Oklahoma. The first forum, held April 23, 2015, had speakers from the Oklahoma Water Resources Board (OWRB), and the Department of Environmental Quality (DEQ). Most of the attendees were either professors or, students, and included those that were proponents for and opponents to water reuse. There was a lack of representation from the public that required more information to develop an opinion regarding IPR. Buyukkamaci and Alkan (2013) conducted a study and found that increased acceptance of water reuse improved with knowledge. Those least likely to approve of water reuse need reassurance that Dave Blue Creek will serve as a reliable environmental buffer. Research of this nature is necessary. Data showing that Dave Blue Creek can increase the quality of reused water will gain the necessary public acceptance to keep moving forward.

It was hypothesized that incubating secondary effluent with sediment would reduce CEC detection via sorption to DBC sediment. Second, the addition of photosynthetically active radiation (PAR) lights would produce a greater decrease in CEC detection by adding photodegradation as an additional mechanism for attenuation. Research has proven that sorption and photodegradation aid in the reduction of CEC in the natural environment (NRC, 2012; Yu et al., 2013; Zhang et al., 2013; Yu and Chu, 2009; Hawker et al., 2011), but the extent to which an environmental buffer helps attenuate CEC is site specific (Hawker et al., 2011). This

was a preliminary study initiated to determine how a diverse group of CEC behaved when incubated with Dave Blue Creek sediment and PAR lights in a laboratory setting. Two microcosm study sets were initiated with treated effluent and DBC sediment to complete this evaluation. Decreases in the number of CEC detected over the course of the experiment would demonstrate DBC's capacity to attenuate CEC as an environmental buffer.

Chapter 3. Methodology

Microcosm studies were setup to assess the effects of sorption and photodegradation. Two-liter Erlenmeyer flasks were filled with treated effluent and sediment fractions less than 0.25 mm with a 2:1 ratio w/v. The flasks were incubated for 15 days on an orbital shaker at 125 rpm. Because of the size of the platform and vessels used, the sorption and photodegradation study sets were ran separately. The sorption experiment had two SED microcosms containing sediment and effluent, and a control microcosm containing effluent only. The microcosms were wrapped in aluminum foil for the duration of the experiment. For photodegradation, there were two PAR + SED microcosms containing sediment and effluent, and a PAR + EFF microcosm containing effluent only. These flasks were irradiated under PAR lights for two hours per day. PAR lights have typical wavelengths of 400–700 nm (Gerbersdorf and Schubert, 2011), the range of visible light. The PAR lights in this experiment were 3100 K warm tone bulbs with 1900 lumens. For sorption, the flasks were wrapped in aluminum foil then placed under a cardboard box for light exclusion. Every five days 80-mL aliquots were extracted from the microcosms and sent off to EEA for analysis.

This research required field sampling and laboratory work. First, sediment samples were collected from DBC near 60th Ave SE (locations shown on Figure 3.1). The sediment samples were wet sieved, and tested for moisture content, loss on ignition, and analyzed for particle size distribution. Second, treated effluent was

sampled at the NWRF after the secondary clarifier. Two 40-mL amber glass vials, two 2-L high density polyethylene (HDPE) bottles, and three 2.5-L amber glass jars were used for collection. The vials were immediately sent off to EEA for preliminary

Figure 3-1 Aerial view of Dave Blue Creek and sediment sampling location in Norman, Oklahoma.

analysis. Effluent in the HDPE was analyzed for nutrient content in the Center for Restoration of Ecosystems and Watersheds (CREW) Laboratory.

3.1 Sediment Sampling

Sediment was sampled near 60th Ave SE along Dave Blue Creek (Figure 3.1). Within the site, three transects were made to obtain a 3-kg composite sediment sample. The sediment was transported and stored in a clean five-gallon bucket secured with a lid. In the laboratory, the sediment was characterized for moisture content, loss on ignition, and particle-size distribution.

3.1.1 Moisture Content

The moisture content of the sediment was measured in accordance with ASTM D2216 (2010). Fifty-three grams of moist sediment was dried in an oven for 16 hours at 110 ± 5 °C. Afterwards, the sediment was transferred into a desiccator to cool. The sample was weighed on an analytical balance, and the mass loss was considered the moisture content of the sample.

3.1.2 Loss on Ignition

Loss on ignition (LOI) followed a modified method described by Ben-Dor and Banin (1989). Two grams (<0.4 mm) of air dried sediment was placed in an oven for 24 hours at 105 °C. Following this, the sample was cooled in a desiccator for 30-minutes before the oven-dry weight was recorded. Then the sediment sample was ignited in a muffle furnace for 16 hours at 440 °C. After ignition, the sediment was

cooled in a desiccator, then evaluated for a final weight (Equation 3-1). Percent LOI was calculated as:

$$\text{LOI (weight \%)} = \frac{\text{Initial weight (g)} - \text{Final weight (g)}}{\text{Initial weight (g)}} \qquad \textbf{Equation 3-1}$$

3.1.3 Sediment Particle Size Analysis

Particle-size analysis used a modified wet sieving procedure of ASTM C92 (2015). The sieves used to obtain the particle size distribution were #10, #60, #100, and #200 (Table 3-1). Three-hundred grams of air dried sediment was mixed in a beaker with deionized (DI) water until the contents formed a slurry. The contents in the beaker were then poured onto the sieves. Any remaining sediment was rinsed with more DI water. Using a rubber spatula, particles retained on the upper most sieve were sprayed with DI water, and gently spread around to work the sediments through to the next sieve. Once all the particles stopped passing through the uppermost sieve, the remaining sediment was carefully scraped into a beaker using deionized water and the spatula. This process was repeated until the sediment retained on each sieve was placed in beakers. These sediments were then dried in an oven at 105 °C until the DI water evaporated. After cooling in a desiccator, the weights of the samples were recorded to 0.1 g. Sediment fractionation was then used to determine sediment texture by referencing a texture triangle.

Table 3-1 Diameters of the particle sizes passing the sieves.

Sieve Number	Diameter Passing (mm)
#10	2.00–0.25
#60	0.25–0.149
#100	0.149–0.075
#200	<0.075

3.2 Treated Effluent

3.2.1 Field Site Description

The NWRF (Figure 3-2) began operating in 1942, with a mission to "produce environmentally safe water at the lowest cost to the citizens of Norman" (NWRF, 2017). Since then, the plant has undergone eight facility upgrades, and services over 92,000 citizens within the municipality. On average, the NWRF treats 15 MGD of influent, with wet weather peaks of 30 MGD. The facility operates under the Oklahoma Pollution Discharge Eliminations System (OPDES), a permit enforced by the ODEQ. This permit prevents the discharge of pollutants, non-storm water, or any matter other than trace amounts that would impair the use of the receiving water (NWRF, 2017).

Figure 3-2 Illustration of the Norman Water Reclamation Facility in relation to Dave Blue Creek.

Upon entry into the NWRF, wastewater influent is pretreated to remove unwanted chemicals and grease (NWRF, 2017). Then the influent goes into the plants headworks where bar screens, conveyors, compactors, and a grit removal system eliminates large and inorganic materials, such as suspended solids, particles and metals, from the influent stream. During primary treatment, floatables and settleable organics are floated and settled out through primary clarifiers and gravity thickeners. The sludge obtained in this stage is treated in an anaerobic digester, and applied to land (NWRF, 2017). In the final stage of treatment, microorganisms, present in activated sludge digest suspended materials in the water. This water is processed through final clarifiers, and polished by UV treatment just before being discharged into the Canadian River, where the highly-treated effluent flows downstream to Lake Eufaula (NWRF, 2017).

3.2.2 Effluent Sampling Protocol

Effluent was grab sampled near the discharge point for the NWRF, and immediately packed on ice. Sampling equipment consisted of three 1200-mL amber glass jars, two 40-mL amber glass vials, powderless nitrile gloves, two 2-L high density polyethylene (HDPE) bottles, and storage coolers. The amber glass jars were triple rinsed with deionized water, autoclaved for 30 minutes at 121 °C, dried in an oven, and stored. Effluent collected in the amber glass jars were set aside for the microcosm studies. HDPE bottles were not previously used and remained in packaging until the sampling event. The HDPE bottles were used for preliminary nutrient analyses back in the CREW Laboratory. The amber glass vials were provided by EEA, and were sampled last to provide the most accurate initial concentrations of CEC. Grab samples were collected using a jar connected to an extended pole. After triple rinsing the jar with effluent, it was repeatedly submerged into the effluent collection box to fill the amber glass jars, vials, and HDPE bottles. All collected samples were labeled with the sampler initials, time, date, and location of sample collection, as well as the sample name. It took two trips to collect the effluent required to complete the study.

3.2.3 Sampler Preparation

Samples collected in amber glassware were being used to determine trace (ng/L) concentrations of CEC, and as such were vulnerable to sample contamination. Measures were taken to avoid contamination from (EEA, 2013):

1. Soaps, detergents, and antibacterial cleansers

2. DEET (active ingredient in insect repellent)
3. Fragrances (cologne, aftershave, perfume)
4. Caffeine or sweeteners (coffee, tea, colas)
5. Prescription drugs, medications, and hormonal substances
6. Over-the-counter medication
7. Antibiotics
8. Tobacco
9. Sunscreen

Powderless nitrile gloves were worn for sampling and processing the effluent, and changed between tasks where cross contamination was a concern. Prior to sampling, personal care products and antibiotics were avoided. On the day of sampling, contact was minimized between clothing and samples and equipment. Furthermore, extra care was taken to not breathe directly in or on samples or collection devices.

3.2.4 Effluent Quality Testing Parameters

Nutrient analyses were performed prior to and after the start of the microcosm studies. Nitrate, nitrite, ammonia, five-day biochemical oxygen demand (BOD) and soluble/total reactive phosphorus were analyzed within 48 hours of the sampling event. Concentrations of total suspended solids (TSS) were measured within the week. Prior to testing, HDPE bottles were brought to room temperature using a water bath. Once the microcosm studies were complete, remaining water was centrifuged, stored in HDPE bottles, and reanalyzed for nitrate, nitrite, soluble/total

reactive phosphorus, and TSS. Dissolved oxygen and pH measurements were obtained from a laboratory technician at the NWRF.

3.2.4.1 Five-day biochemical oxygen demand

This test followed the procedure outlined by SM 5210B (SM, 2001). Twenty-four hours prior to sampling, dilution water was prepared in a nine-liter carboy with deionized water and nutrient pillows. The carboy was stirred and aerated overnight, and remained this way throughout the procedure. Once the samples were brought to room temperature, Polyseed® water was made by depositing the contents of a Polyseed® capsule in 500 mL of dilution water. The Polyseed® was stirred and aerated for an hour, decanted into a new beaker, and placed back on the stir plate with an air stone. From these solutions, four Polyseed® controls, three glucose glutamic acid (GGA) samples, and two blank controls were prepared. BOD sample bottles were filled halfway with sample water, 0.16 g of nitrification inhibitor and 4 mL of Polyseed® was added, then the bottle was filled with effluent. Initial DO concentrations were measured for 3–5 minutes after preparation. All samples were filled to the neck of the BOD bottle, stoppered, and capped to ensure air bubbles were not present. The BOD bottles were incubated at 20 °C for five days. After 120 hours, the final DO concentrations were measured. The initial and final DO readings were then used to calculate the BOD_5 of the effluent.

3.2.4.2 Nitrate-N

Nitrate-nitrogen concentrations were measured by using the Hach Dimethylphenol Method 10206, approved by 40 CFR 141 (Hach, 2015a). Test-n-tube kits (TNT 835/836) were used in accordance with the method, and came equipped with test tube vials and a bottle of Solution A. Sample water was placed in a test tube vial containing 2,6-dimethylphenol (Hach, 2015a). The contents were reacted with solutions containing sulfuric and phosphoric acids (Solution A) to form 4-nitro-2,6-dimethylphenol, which produces a colored solution based on the amount of nitrate ions present. Once Solution A was added to the test vial, the contents were inverted until thoroughly mixed. The test vial was then placed on the benchtop to allow a 15-minute reaction to occur, before being analyzed in a DR 3800 spectrophotometer. Each test-n-tube (TNT) vial comes with a barcode that allows the machine to measure the results under the proper method and wavelength, 345 nm (Hach, 2015a). The light source penetrates the solution capturing 10 measurements in one rotation to exclude outliers or flawed data (Hach, 2016).

3.2.4.3 Nitrite-N

Nitrite-nitrogen concentrations were quantified by using a Hach Diazotization Method 10207, an equivalent method for EPA 353.2 (Hach, 2014a). Test-n-tube kits (TNT 839/840) were used in accordance with the method, and came equipped with test tube vials and DosiCap™ Zip caps. The vials contained a solution of N-(1-naphthyl)-ethylenediamine dihydrochloride, topped with zip caps containing sulfanilamide reagent powder. Nitrite ions present in the water sample reacted with a

primary aromatic amine in acidic solution which formed a diazonium salt (Sreekumar et al., 2003). During a reaction time of 10 minutes, a complex azo dye formed that was directly proportional to the amount of nitrite in the sample (Hach, 2014a). Then the vial was placed in the appropriate slot in a DR 3800 spectrophotometer. The machine read the barcode on the vial to select the proper method and wavelength, 515 nm, for the light source, and produced a reading.

3.2.4.4 Ammonia-N

Ammonia-nitrogen content was determined by using the Hach Salicylate Method 10205, an equivalent method for EPA 350.1 (Hach, 2015b). The ammonia TNT kit was equipped with test tube vials and DosiCap™ Zip caps. In this process ammonium ions interact with hypochlorite and salicylate ions in the presence of the sodium nitroprusside solution in the TNT ammonia vials (Hach, 2015b). After 5-mL of sample water was added to the vials, a zip cap containing an ammonium salicylate reagent was added to the solution, which formed, indophenol blue over a 15-minute reaction period. This blue color intensified with the amount of ammonia-nitrogen present in the water sample. Then the vials were analyzed in the DR 3800 spectrophotometer at the end of the15-minute reaction period. The vial was placed in the appropriate slot in the machine. Once the barcode on the vial was read to select the proper method and wavelength, 694 nm, for the light source, and produced an ammonia-nitrogen reading.

3.2.4.5 Total and Soluble Reactive Phosphorus

Total (TRP) and soluble reactive phosphorus (SRP) were measured using the Hach Ascorbic Acid Method 10209, an equivalent method for EPA 365.1 (Hach, 2014b). Phosphorus TNT kits were equipped with vials containing ascorbic acid (Solution B). Two milliliters of effluent and the 0.2 mL of Solution B (ammonium molybdate and antimony potassium) were added to the test vial. The vial contents and Solution B reacted to form an antimony-phosphate-molybdate complex for a 10-minute reaction time (Hach, 2014b). The intensity of the solution color was directly proportional to the phosphorus concentration. After the 10-minute reaction time, the vial was placed in the DR 3800 spectrophotometer. The barcode signaled the machine to use the proper method and wavelength, 880 nm, for the light source, and produced a phosphorus reading. SRP analyses required the sample water to be filtered through Becton Dickinson medical syringes with Whatman 0.45-µm nylon filter caps, prior to placing the water in the vials. Then the tests were completed as above in the TRP analyses.

3.2.4.6 Total Suspended Solids

The non-filterable residue was determined using the procedure outlined in EPA 160.1 (EPA, 1971). A suction flask fitted with the appropriate stopper was connected to a vacuum pump. Gelman type A/E 4.7-mm glass fiber filters were placed into the bottom of a Gooch crucible, then flushed with three successive 20-mL volumes of deionized water. Washings were discarded and 100-mL of well mixed sample water was suctioned through the crucible. The filtered sample was

transferred to a drying dish and evaporated in an oven at 105 °C for an hour, cooled in a desiccator, and weighed. The drying cycle was repeated until a constant weight of 0.5 mg or less was achieved. The non-filterable residue was calculated using Equation 3-2:

$$\text{Non-filterable residue (mg/L)} = \frac{(A\text{-}B)*1000}{C} \qquad \textbf{Equation 3-2}$$

where:

A = weight of filter and crucible + residue in mg

B = weight of filter and crucible in mg

C = mL of sample filtered

3.2.4.7 CEC Analytical Methods and Equipment

A suite of 98 CEC (Table 3-2) was analyzed using EPA Methods 539 and 1964. These methods were validated for their accuracy and precision of results by the Water Research Foundation (Jones, 2016). Analyses were performed using positive or negative mode electrospray ionization (+/-ESI) LC-MS-MS (Eaton and Haghani, 2012). For the subscribed methods, each analyte was directly infused into the LC-MS-MS with multiple mass transitions, and concentrated onto a solid phase extraction column (Eaton and Haghani, 2012). Target analytes were refocused on an analytical column, separated, and eluted into a mass spectrometer (Eaton and Haghani, 2012). Acidic and basic eluents for the positive and negative modes were then used to create sensitivity on the mass spectrometer (Eaton and Haghani, 2012). Analytes were pinpointed based on their affinity to protonate or deprotonate in +/-

ESI modes. If the measured mass intensity of a CEC ionized adduct specie trended towards ESI+, then the compound contained nitrogen. If the ionized adduct specie trended towards ESI-, then the compound contained a carboxylic group (Eaton and Haghani, 2012). These trends helped delineate the specific analytes.

3.3 Microcosm Studies

The studies consisted of mixing NWRF treated effluent, and DBC sediment on a MaxQ 2000 orbital shaker. Each study set began within 10-hours of effluent collection. Sample water not used immediately so it was brought to room temperature prior to use. The sediment was wet sieved pass #60 (<0.25 mm) sieve, dried, and ground with a mortar and pestle. All vessels used for the experimental setup and extraction were autoclaved at 121 °C for 30 minutes and dried at 50 °C. Since CEC concentrations were low the same care required during sample preparation was employed throughout the study as well.

Table 3-2 List of 98 CEC investigated in this project categorized by classification.

Compound	Classification	Sub-classification
4 nonylphenol (semi-quantitative)	EDC	Surfactant
4-tert-Octyphenol	EDC	Surfactant
Androstenedione	EDC	Steroid hormone
Bisphenol-A	EDC	Plasticizer
Estradiol	EDC	Estrogen hormone
Estriol	EDC	Estrogen hormone
Estrone	EDC	Estrogen hormone
Ethinyl Estradiol-17 Alpha	EDC	Contraceptive
Norethisterone	EDC	Steroid hormone
Progesterone	EDC	Steroid hormone
Testosterone	EDC	Male hormone
Acetaminophen	PPCP	Analgesic
Albuterol	PPCP	Anti-asthmatic
Amoxicillin (semi-quantitative)	PPCP	Antibiotic
Atenolol	PPCP	Cardio
Azithromycin	PPCP	Antibiotic
Bendroflumethiazide	PPCP	Anti-hypertension
Bezafibrate	PPCP	Cardio
Butalbital	PPCP	Analgesic
Carbadox	PPCP	Antibiotic
Carbamazepine	PPCP	Anti-seizure
Carisoprodol	PPCP	Muscle relaxer
Chloramphenicol	PPCP	Antibiotic
Cimetidine	PPCP	Cardio
Dehydronifedipine	PPCP	Cardio
Diazepam	PPCP	Anti-anxiety
Diclofenac	PPCP	Anti-inflammatory
Dilantin	PPCP	Anti-seizure
Diltiazem	PPCP	Blood pressure
Erythromycin	PPCP	Antibiotic
Flumequine	PPCP	Antibiotic
Fluoxetine	PPCP	Antidepressant
Gemfibrozil	PPCP	Cardio
Ibuprofen	PPCP	Analgesic
Iohexal	PPCP	X-ray contrast
Iopromide	PPCP	X-ray contrast

Table 3-2 (Continued).

Compound	Classification	Sub-classification
Ketoprofen	PPCP	Anti-inflammatory
Ketorolac	PPCP	Anti-inflammatory
Lidocaine	PPCP	Analgesic
Lincomycin	PPCP	Analgesic
Lopressor	PPCP	Cardio
Meclofenamic Acid	PPCP	Anti-inflammatory
Meprobamate	PPCP	Anti-anxiety
Naproxen	PPCP	Analgesic
Nifedipine	PPCP	Cardio
Oxolinic Acid	PPCP	Antibiotic
Pentoxifylline	PPCP	Blood thinner
Phenazone	PPCP	Analgesic
Primidone	PPCP	Anti-seizure
Salicylic Acid	PPCP	Antibacterial
Sulfachloropyridazine	PPCP	Antibiotic
Sulfadiazine	PPCP	Antibiotic
Sulfadimethoxine	PPCP	Antibiotic
Sulfamazerine	PPCP	Antibiotic
Sulfamethazine	PPCP	Antibiotic
Sulfamethizole	PPCP	Antibiotic
Sulfamethoxazole	PPCP	Antibiotic
Sulfathiazole	PPCP	Antibiotic
Theophylline	PPCP	Anti-asthmatic
Warfarin	PPCP	Cardio
1,7- Dimethylxanthine	Stimulant	Caffeine metabolite
Caffeine	Stimulant	Caffeine
Cotinine	Stimulant	Nicotine metabolite
Theobromine	Stimulant	Caffeine metabolite
Butylparaben	Preservative	Anti-microbial
Ethylparaben	Preservative	Antifungal
Isobutylparaben	Preservative	Antibacterial/fungal
Methylparaben	Preservative	Antibacterial/fungal
Propylparaben	Preservative	Antibacterial/fungal
Thiabendazole	Preservative	Antibacterial/fungal
Triclocarban	Preservative	Antibacterial
Triclosan	Preservative	Antibacterial
Trimethoprim	Preservative	Antibacterial

Table 3-2 (Continued).

Compound	Classification	Sub-classification
Acesulfame-K	Sweetener	Sugar substitute
Sucralose	Sweetener	Sugar substitute
2,4- D	Pesticide	Herbicide
Atrazine	Pesticide	Herbicide
Bromacil	Pesticide	Herbicide
Chloidazon	Pesticide	Herbicide
Chlorotoluron	Pesticide	Herbicide
Clofibric Acid	Pesticide	Herbicide
Cyanizine	Pesticide	Herbicide
DACT	Pesticide	Atrazine metabolite
DEA	Pesticide	Atrazine metabolite
DEET	Pesticide	Mosquito repellant
Diazepam	Pesticide	Atrazine metabolite
Diuron	Pesticide	Herbicide
Isoproturon	Pesticide	Herbicide
Linuron	Pesticide	Herbicide
Metazachlor	Pesticide	Herbicide
Metolachlor	Pesticide	Herbicide
OUST (Sulfameturon methyl)	Pesticide	Herbicide
Propazine	Pesticide	Herbicide
Quinoline	Pesticide	Herbicide feedstock
Simazine	Pesticide	Herbicide
TCEP	Flame Retardant	Fabric coating
TCPP	Flame Retardant	Fabric coating
TDCPP	Flame Retardant	Fabric coating

Sources: Jones, 2016; Laubacher 2016; NRC, 2012; AWWA, 2007

3.3.1 Experiment Setup

These microcosm studies were the crux of this research. There were two study sets used to assess the effects of sorption + photodegradation (PAR + SED 2x, PAR + Effluent) and sorption (SED 2x, Effluent). Fewer detections, over the duration of the microcosm study, were expected for CEC that were more susceptible to sorption. Similarly, the number of detections of CEC more prone to

photodegradation would decrease from the initial day (day 0) to the end (day 15) of the sorption + photodegradation incubation period.

Each study was incubated for 15 days, the estimated hydraulic retention time of DBC, from 36th Ave SE to Lake Thunderbird. The estimated HRT was determined by using the parameters in Table 3-4 and the proposed discharge of 6 MGD (Carollo, 2014), into Dave Blue Creek. The volume was calculated by dividing the average cross sectional areas at 48th, 60th, and 72nd (Figure 3-3) and the length of the creek (Table 3-3). With the volume and discharge rate, the hydraulic retention time (HRT) was estimated at ~16 days. Since three aliquot extractions were planned over the course of the experiment, an HRT of 15 days was chosen for equivalent incubation times between extractions.

Table 3-3 Parameters used to determine the hydraulic retention time of Dave Blue Creek.

HRT Parameters	
Length (ft)	30244
Depth (ft)	8
	12
	14
Width (ft)	18
	50
	45

Figure 3-3 Aerial view of Dave Blue Creek and locations where cross-sectional area (brown triangles) measurements were taken.

3.3.1.1 PAR + Sediment Microcosm Study Set

Experiments run under PAR lights represented sunlight in the natural environment (Figure 3-4). DBC is surrounded by heavy vegetation, thus minimal sunlight exposure was simulated in the experiment. PAR + SED microcosms contained sediment and effluent, and the PAR + Effluent microcosm contained only effluent. Each vessel was sealed with parafilm, and irradiated under PAR lights for two hours a day. Increased sunlight exposure is expected in DBC, but the experiment irradiation time was appropriate to model a worst-case scenario for CEC attenuation.

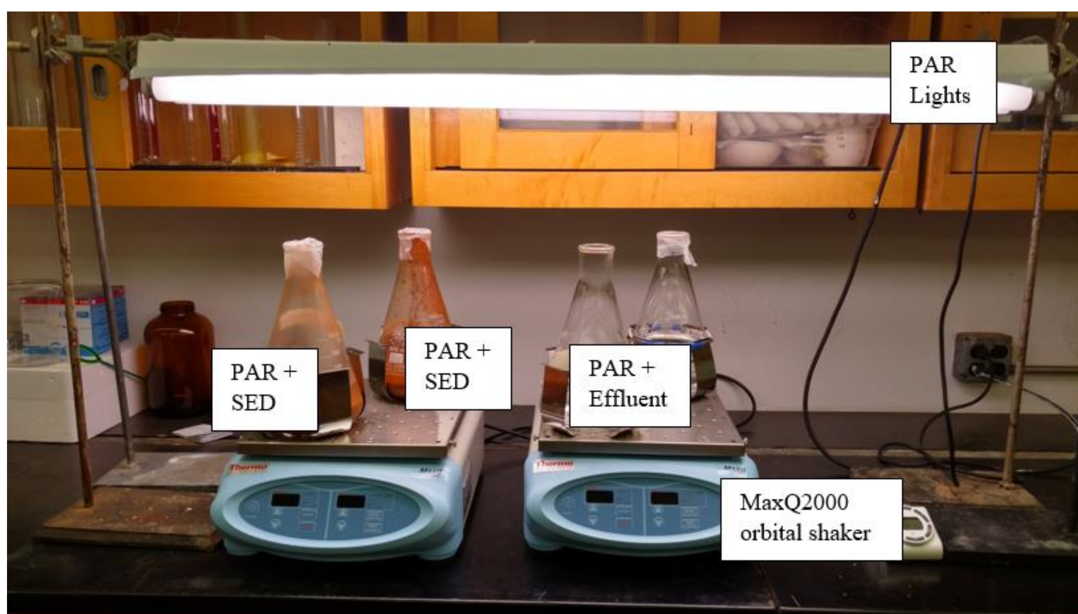

Figure 3-4 PAR + SED microcosm setup: PAR + SED, PAR + SED duplicate, and PAR + Effluent.

3.3.1.2 Sediment Microcosm Study Set

Experiments run without PAR lights were used to assess whether CEC detection changed with incubation of DBC sediment. SED experiments contained sediment and effluent, and the control contained effluent only. The flasks were sealed with parafilm, then wrapped in aluminum foil (Figure 3-5). For further light exclusion, the orbital shakers and microcosms were placed under a cardboard box.

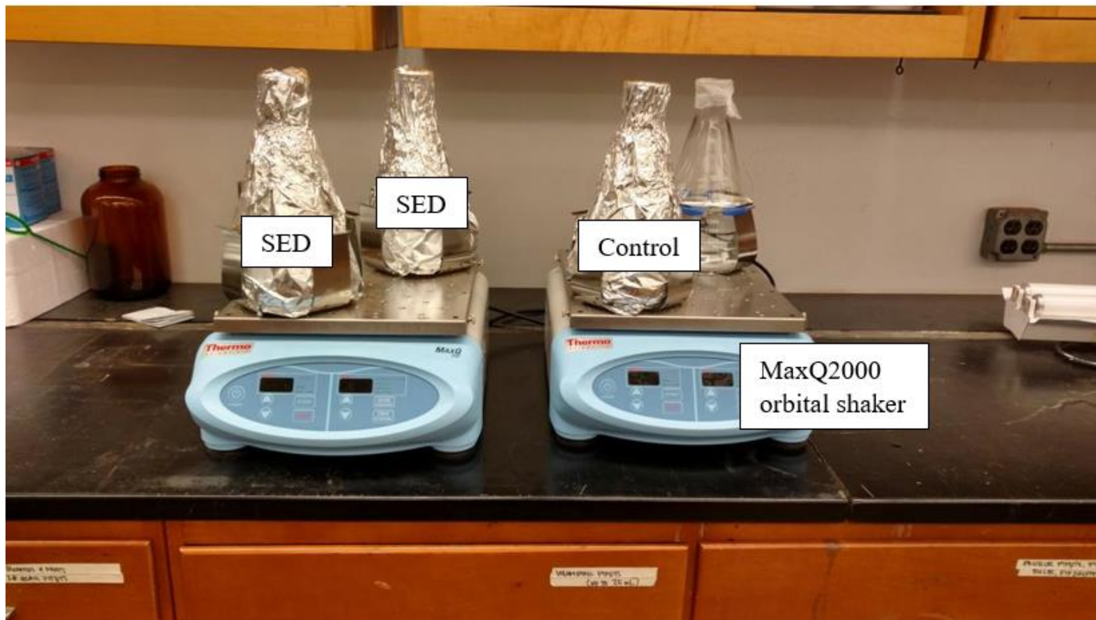

Figure 3-5 Sediment microcosm setup: SED, SED duplicate, and Control; pictured without cardboard box.

3.3.1.3 Sample Extraction

Every fifth day the shakers were stopped for CEC sampling. Eighty milliliters of aliquot were extracted from each flask and transferred into plastic centrifuge tubes. Flasks were recovered, and the tubes were placed in a Beckman J2-21 centrifuge and spun for 10 minutes at 5000 rpm. Supernatant was poured into amber glass vials, packed on ice, and sent off to EEA. All samples were prepared using non-powdered sterile nitrile gloves. For the SED and control studies sample extraction was performed in darkness.

3.4 QAQC

During the sampling events a field duplicate was collected for nutrient analyses and microcosm studies. Once in the laboratory, nutrient analyses in each

study set were tested along with a laboratory blank, laboratory duplicate, sample spike, and laboratory fortified blank to assess the quality and accuracy of laboratory work. The blanks consisted of deionized water from the laboratory. Duplicate samples were retested for nutrients from a bottle collected in the field. The relative percent difference (%RPD) was calculated for each analyte and compared to the laboratory and field duplicates. Acceptable %RPD values were less than or equal to 20%. Spikes were samples containing a known concentration and volume of a standard solution, and the laboratory fortified blanks (LFB) were diluted standard solutions prepared and tested like the sample set. Acceptable recovery limits were used to test the accuracy of the spikes and LFBs. The acceptable recovery limits for spikes and LFBs, were 75–125% and 90–110%, respectively.

3.5 Microcosm Data Analysis

The number of CEC detected initially in the effluent and on the final day of the microcosm study were used to calculate percent reductions for the microcosm study sets. For an in depth look at preferred pathways, percent reduction calculations were performed on the concentrations of specific analytes. When analyte detections were below the MRL (Table 3-4), the MRL was substituted as the day 15 concentration, and percent reductions reported as greater than the calculated value.

Table 3-4 List of CEC minimum reporting limits for individual analytes.

Compound	MRL (ng/L)
4 nonylphenol (semi-quantitative)	100
4-tert-Octyphenol	50
Androstenedione	5
Bisphenol-A (BPA)	10
Estradiol	5
Estriol	5
Estrone	5
Ethinyl Estradiol- 17 Alpha	5
Norethisterone	5
Progesterone	5
Testosterone	5
Acetaminophen	5
Albuterol	5
Amoxicillin (semi-quantitative)	20
Atenolol	5
Azithromycin	20
Bendroflumethiazide	5
Bezafibrate	5
Butalbital	5
Carbadox	5
Carbamazepine	5
Carisoprodol	5
Chloramphenicol	10
Cimetidine	5
Dehydronifedipine	5
Diazepam	5
Diclofenac	5
Dilantin	20
Diltiazem	5
Erythromycin	10
Flumequine	10
Fluoxetine	10
Gemfibrozil	5
Ibuprofen	10
Iohexal	10
Iopromide	5

Table 3-4 (Continued).

Compound	MRL (ng/L)
Ketoprofen	5
Ketorolac	5
Lidocaine	5
Lincomycin	10
Lopressor	20
Meclofenamic Acid	5
Meprobamate	5
Naproxen	10
Nifedipine	20
Oxolinic Acid	10
Pentoxifylline	5
Phenazone	5
Primidone	5
Salicylic Acid	100
Sulfachloropyridazine	5
Sulfadiazine	5
Sulfadimethoxine	5
Sulfamazerine	5
Sulfamethazine	5
Sulfamethizole	5
Sulfamethoxazole	5
Sulfathiazole	5
Theophylline	20
Warfarin	5
1,7- Dimethylxanthine	10
Caffeine	5
Cotinine	10
Theobromine	10
Butylparaben	5
Ethylparaben	20
Isobutylparaben	5
Methylparaben	20
Propylparaben	5
Thiabendazole	5
Triclocarban	5
Triclosan	10
Trimethoprim	5

Table 3-4 (Continued).

Compound	MRL (ng/L)
Acesulfame-K	20
Sucralose	100
2,4- D	5
Atrazine	5
Bromacil	5
Chloidazon	5
Chlorotoluron	5
Clofibric Acid	5
Cyanizine	5
DACT	5
DEA	5
DEET	10
Diazepam	5
Diuron	5
Isoproturon	100
Linuron	5
Metazachlor	5
Metolachlor	5
OUST (Sulfameturon methyl)	5
Propazine	5
Quinoline	5
Simazine	5
TCEP	10
TCPP	100
TDCPP	100

Source: Eurofins Eaton Analytical, Inc.

Chapter 4. Results and Discussion

4.1 Microcosm Water Quality

Sediment used in the microcosms was a loamy-sand with an organic matter content of ~1%. Initial alkalinity and turbidity for the PAR + SED and PAR + Effluent studies were 116 mg/L $CaCO_3$ and 6.65 NTU, respectively. For the SED and control studies the alkalinity and turbidity readings were 135 mg/L $CaCO_3$ and 8.83 NTU, respectively. Nitrate-N, nitrite-N, ammonia-N, and (soluble) orthophosphorus readings were taken before and after incubation in the microcosms (Appendix A).

4.2 CEC Detection Results

There were 40 and 32 CEC detections, out of 98 analytes at the beginning of the PAR + SED and SED experiment sets, respectively. Number of CEC detections varied across the microcosms (Table 4-1). Despite increased CEC detects on days 5 or 10 in the PAR + SED duplicate, PAR + Effluent, SED, and control microcosms, analyte detections still decreased from initial to final (day 15) sampling events.

Table 4-1 Summary of CEC detections during each period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	40	33	33	16
PAR + SED	40	34	43	17
PAR + Effluent	40	47	31	30
SED	32	22	32	20
SED	32	27	35	18
Control	32	27	43	30

CEC detects in the control microcosm illustrate that incubating secondary effluent alone will produce a small reduction in CEC detections. The control experiment had two fewer chemical constituents detected on day 15 than initiation of the microcosm study (day 0). By incubating secondary effluent under PAR lights (e.g., PAR + Effluent microcosm) the number of detections decreased more notably as opposed to detections in the control. The addition of sediment to effluent in the SED microcosms also decreased the CEC detects by the final day of experimentation. Further, the additive effects of both PAR and sediment on effluent (e.g., PAR + SED) resulted in an even higher reduction of CEC detections. These observations suggest that environmental buffers possess qualities that can decrease the number of CEC analytes detected in secondary effluent. In the following sections, CEC detects will be assessed by classification to determine what effects apparent sorption and photodegradation had on the different CEC classes.

4.2.1 Endocrine Disrupting Compounds

EDCs contain the third greatest number of compounds analyzed for the microcosm studies. There were four EDC detects in the microcosms out of 11 (Table 4-2). A near constant decrease in EDC detects was observed over the course of the experiment. Detections ranged from zero to two by day 15, meaning EDCs quantified had reductions greater than or equal to half the initial detects. Comparison of the PAR + SED, PAR + Effluent, and SED microcosms suggest that both sediment and PAR lights aid in the reduction of EDCs.

Table 4-2 Summary of EDC detections during each period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	4	2	4	1
PAR + SED	4	2	2	1
PAR + Effluent	4	3	2	1
SED	4	3	2	2
SED	4	4	2	0
Control	4	4	2	0

The most frequently detected EDC in the microcosm studies was 4-nonylphenpl (4NP) (Figure 4-1). Concentrations of 4NP increased in the microcosms except for the SED experiment. This may be attributed to two factors: residual 4NP present on the flasks, and persistence of 4NP in sediment. The flasks were cleaned with deionized water and Liquinox® detergent, autoclaved, and dried prior to beginning the study. Because 4NP is a surfactant metabolite, it could have remained in the flasks in trace amounts, increasing 4NP concentrations in the microcosms. Because of a high octanol-water partition coefficient (K_{ow} = 5.76) (Hawker et al., 2011), 4NP may have been sorbed to the sediment that was collected from DBC. Background concentrations of CEC in sediment was not assessed in this research and the sediment half-life of 4NP is 135 days (Hawker et al., 2011). Disturbance of the sediment-water interface could have caused desorption of 4NP from the sediment during extractions. Incubation time was 15 days with 30 cumulative hours of PAR

radiation. Complete photodegradation may not have occurred because the microcosm study was 15 days and 4NP has a photodegradation half-life of 7.4 days (Hawker et al., 2011). However, the decrease in the number of detections indicates that photodegradation and sorption played a role in the reduction of 4NP concentrations (Figure 4-1). Further, 4NP concentrations of <5 ng/L on day 15 in the SED and control microcosms suggest that the analyte can also degrade without sediment or PAR (Hawker et al. 2011).

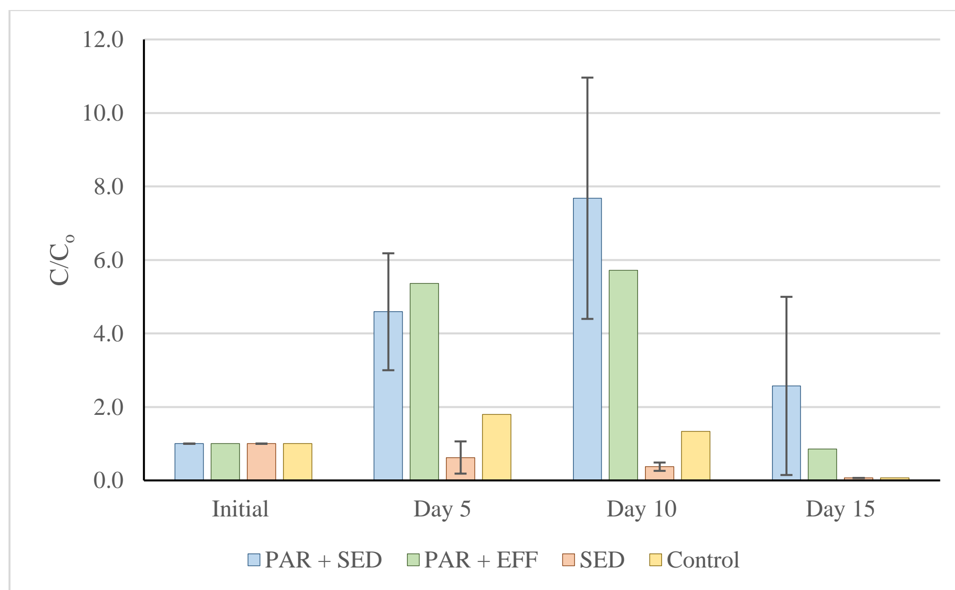

Figure 4-1 Relative change in concentration for 4-nonylphenol in each microcosm; PAR + SED and SED error bars are means ± one standard deviation in duplicate; SED and control concentrations on day 15 are <100 ng/L.

4.2.2 Pharmaceuticals and Personal Care Products

PPCPs represent half of the CEC analyzed in the experiment. There were 21 PPCPs detected in the microcosms incubating under PAR lights, and 10 in the microcosms without PAR lights (Table 4-2). PPCP detects increased on day 10 in the

PAR + SED, SED, and control microcosms. Sediment appeared to be less effective in decreasing PPCP detection than PAR by the end of the studies. When the effluent was irradiated under PAR lights, there was an observed decrease in PPCP detects. There were seven fewer detections in the PAR + Effluent microcosm by day 15. The collective effects of PAR and sediment resulted in the fewest CEC detects by day 15. Detections decreased by two-thirds or more in the PAR + SED microcosms. PPCP detects appear to be more influenced by incubation under PAR lights than interaction with DBC sediment alone, suggesting photodegradation plays a role in the reduction of PPCP detects in environmental buffers.

Table 4-3 Summary of PPCP detections during each period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	21	15	17	5
PAR + SED	21	17	22	7
PAR + Effluent	21	21	16	14
SED	10	9	14	9
SED	10	10	16	10
Control	10	10	22	16

Lidocaine (LDC) is a highly-prescribed analgesic (Ruá-Gómez and Püttman, 2013), which contributes to its persistence in the aquatic environment. Despite increased LDC concentrations in the control and PAR + SED microcosm on days 5 and 10, respectively, the positive effects of sorption and photodegradation was observed in the microcosms (Figure 4-2). Ruá-Gómez and Püttman (2013)

discovered sunlight-induced hydroxyl radicals were the main cause of LDC photodegradation, whereas direct sunlight slowed the degradation of LDC. The PAR + SED, PAR + Effluent, SED microcosms had 4.4%, 9.2%, and 11.2%, respectively, LDC remaining in the microcosms after 15 days. Photodegradation and sorption both appear to serve as pathways for LDC attenuation in this research.

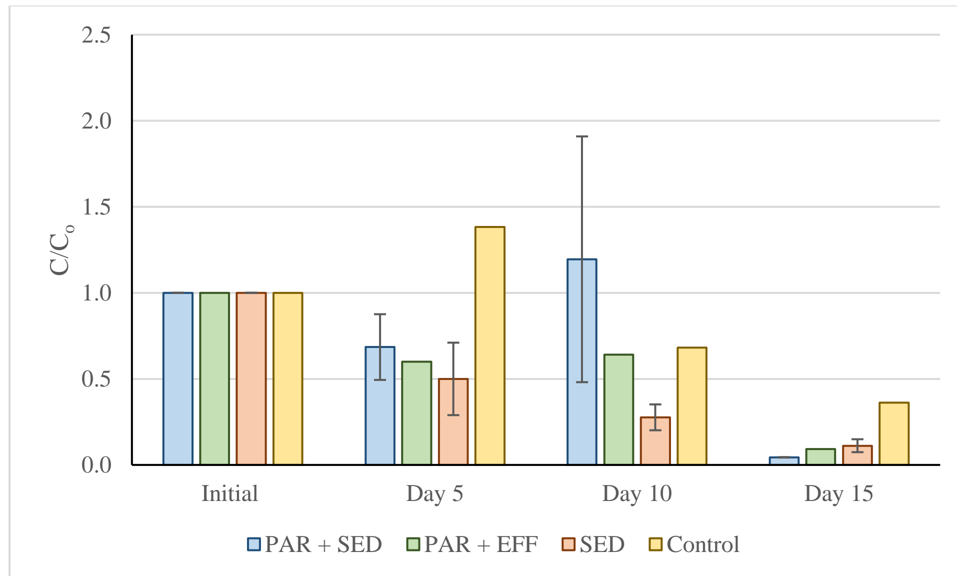

Figure 4-2 Relative change in concentration for lidocaine in each microcosm; PAR + SED and SED error bars are means ± one standard deviation in duplicate.

4.2.3 Stimulants

Stimulants were represented by four compounds in this research. Each microcosm had two initial detections, had varied detection increases and decreases on days 5 and 10, and went back down to one or two stimulants by the end of the study (Table 4-3). Based on detection behavior considerable differences are not observed when stimulants are incubated with sediment or PAR lights.

Table 4-4 Summary of stimulant detections during each period in the microcosms.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	2	3	3	2
PAR + SED	2	2	4	1
PAR + Effluent	2	4	3	1
SED	2	1	2	2
SED	2	1	3	2
Control	2	1	3	1

Theobromine, a caffeine metabolite, was more persistent in the microcosms than its parent compound. Theobromine and another caffeine metabolite, 1,7-Dimethylxanthine, were initially detected in the PAR + SED and PAR + Effluent microcosms. Theobromine remained above MRL (>10 ng/L) throughout the study, while 1,7-dimethylxanthine had concentrations <10 ng/L after 5 days in the microcosms. Caffeine and theobromine were detected in the SED and control microcosms. Theobromine persisted after caffeine concentrations were detected <5 ng/L. Little data were found on the fate of theobromine in the natural environment, but incubation with sediment appeared to be a mechanism for attenuation (Figure 4-3), despite overarching detection data for stimulant detections.

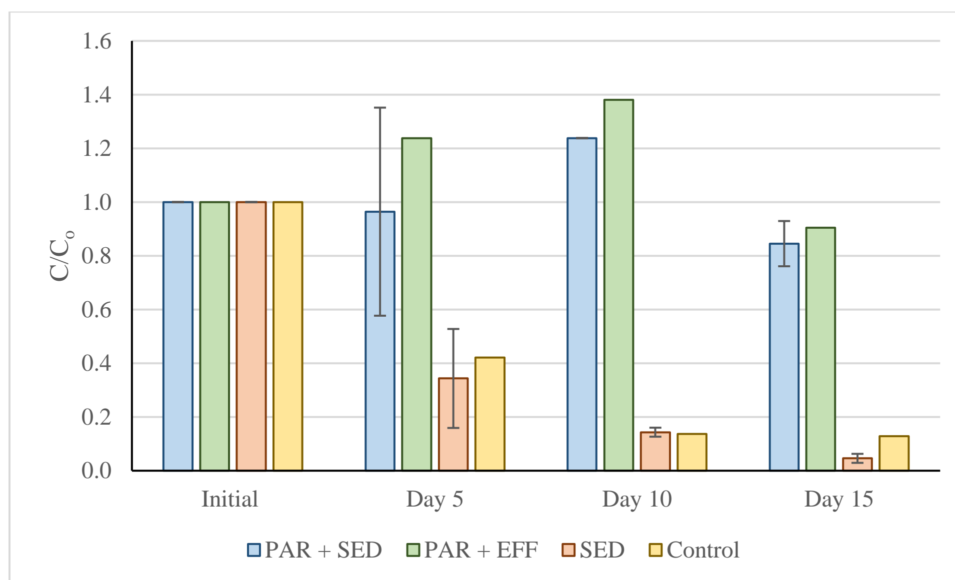

Figure 4-3 Relative change in concentration for theobromine in each microcosm; PAR + SED and SED error bars are means ± one standard deviation in duplicate.

4.2.4 Preservatives

There were two preservatives initially detected in the microcosms (Table 4-4). The PAR + SED and PAR + Effluent had increased CEC detects on day 10 and day 5, respectively, but there were fewer detections in other microcosms as the experiment progressed. There were no preservatives observed above MRL in the SED microcosms and PAR + SED duplicate by day 15, which indicates that sorption and photodegradation occurred during the 15-day period.

Table 4-5 Summary of preservative detections during each period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	2	1	1	1
PAR + SED	2	1	2	0
PAR + Effluent	2	3	1	1
SED	2	1	1	0
SED	2	1	1	0
Control	2	2	2	1

Triclosan (TCS) was initially detected at 22 ng/L in the PAR + SED and PAR + Effluent microcosms (Figure 4-4), but had concentrations <5 ng/L for the duration of the SED and control experiments. TCS has a photodegradation half-life of 81.6 hours (Wu et al., 2015), a log K_{ow} of 4.76 (AWWA, 2007). TCS concentrations in the PAR + SED microcosm were detected below MRL (<5 ng/L) on day 5, whereas an additional sampling period was needed for TCS to fall below 5 ng/L in the PAR + Effluent microcosm. Both mechanisms demonstrate the capacity to reduce TCS concentrations, but the additive effects of photodegradation and sorption appear to be more effective than photodegradation alone.

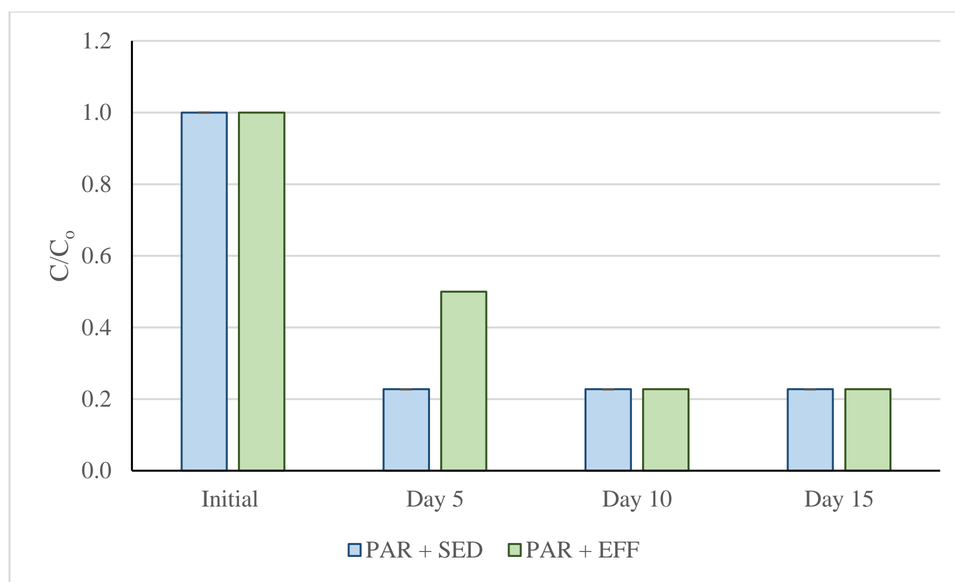

Figure 4-4 Relative change in concentration for triclosan in the PAR + SED and PAR + Effluent microcosms; Concentrations on days 10 and 15 are <5 ng/L.

4.2.5 Artificial Sweeteners

This CEC class is known to be ubiquitous and persistent in the environment (Sang et al., 2014; Perkola et al., 2016). Acesulfame-K and sucralose were the only analytes for this class of CEC. Acesulfame-K was detected below <20 ng/L in the PAR + SED and PAR + Effluent experiments on day 10, causing the number of detects to temporarily decrease (Table 4-5). However, both analytes were detected for most the experiment.

Table 4-6 Summary of sweetener detections during each extraction period in the microcosm studies

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	2	2	1	2
PAR + SED	2	2	2	2
PAR + Effluent	2	2	1	2
SED	2	2	2	2
SED	2	2	2	2
Control	2	2	2	2

Sucralose was consistently detected in the PAR + SED, SED, PAR + Effluent and control microcosms, affirming its persistence in the natural environment (Sang et al., 2014; Batchu et al., 2013). Batchu et al., (2013) investigated the effects of Sun Test lights (300–800 nm) on the photodegradation of sucralose. Sucralose persisted in the study after one month of constant irradiation, and was degraded <16%, leading to the conclusion that natural conditions were insufficient for the degradation of sucralose (Batchu et al., 2013). Despite environmental persistence, sucralose concentrations did decrease under simulated sunlight in this research (Figure 4-5). Initial sucralose concentrations were 42,000 ng/L in the PAR + SED and PAR + Effluent microcosms, and 180,000 ng/L in the SED and control microcosms. Remaining sucralose was 20–40% of the initial values, but the effects of sediment and PAR incubation was observed in the study. This suggests that persistence of sucralose in the natural environment is time dependent. The duration of this study, and the irradiation time were not long enough to observe concentrations of sucralose fall below its MRL (<100 ng/L).

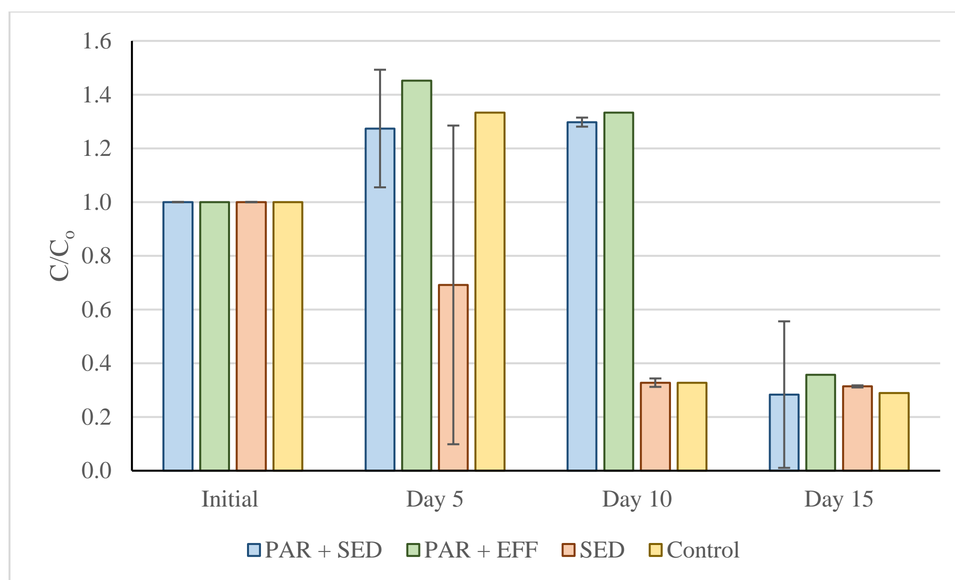

Figure 4-5 Relative change in concentration for sucralose in each microcosm; PAR + SED and SED error bars are means ± one standard deviation in duplicate.

4.2.6 Pesticides

There were 20 pesticides in the CEC suite of analytes. Six were initially detected in the PAR + SED and PAR + Effluent microcosms, and nine in the SED and control studies. Pesticide detection in microcosms incubating under PAR lights varied on days 5 and 10, decreased on day 15. The PAR + Effluent experiment was an exception, and had two additional pesticides observed above MRL. The SED experiments also varied in detection on days 5 and 10, but had five fewer compounds detected on day 15 than at the start of the experiment. The data (Table 4-6) indicates that incubation with sediment was more effective in reducing pesticide detections, than PAR lights and cumulative effects of PAR light and sediment.

Table 4-7 Summary of pesticide detections during each extraction period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	6	8	6	4
PAR + SED	6	2	8	5
PAR + Effluent	6	11	6	8
SED	9	3	10	4
SED	9	6	9	4
Control	9	6	9	7

4.2.7 Flame Retardants

Flame retardants were the second smallest number of CEC monitored in the microcosm studies. Tris(2-carboxylethyl) phosphine (TCEP), tris(1-chloro-2-propyl) phosphate (TCPP), and tris(,3-dichloro-2-propyl) phosphate (TDCPP) were detected in each microcosm (Table 4-7), and are known for their persistence in the environment (AWWA, 2007). In the PAR + Effluent and control the flame retardants were persistent. Detections in the PAR + SED and SED microcosms decreased throughout the experiment. The sole effects of PAR lights on flame retardant detections appear to be insufficient in reducing detections. However, incubation with sediment demonstrated some capacity of the sediments to decrease flame retardant detections, suggesting that sorption may serve as pathway for decreasing the number of flame retardants observed in environmental buffers.

Table 4-8 Summary of flame retardant detections during each extraction period in the microcosm studies.

Microcosm	Initial	Day 5	Day 10	Day 15
PAR + SED	3	2	1	1
PAR + SED	3	2	3	1
PAR + Effluent	3	3	2	3
SED	3	3	1	1
SED	3	3	2	0
Control	3	2	3	3

Chapter 5. Conclusion

De facto water reuse has been a common practice in the US for quite some time (NRC, 2012). Water treatment facilities constantly discharge effluent, and municipalities downstream treat the water and distribute it to the population. CEC are frequently detected at trace concentrations (ng/L) in the natural environment because water treatment facilities are no designed for CEC removal. Water scarcity events, increasing populations, climatic change, and the unpredictability of the magnitude and location of rain events are going to increase the need for understanding CEC removal in wastewater effluents. The suite of analytes studied in this research have different physiochemical properties that cause them to be attenuated by various mechanisms.

This research used sediment from DBC and highly treated effluent from the NWRF to set up microcosm studies. These experiments were incubated for 15 days on orbital shakers at 125 rpm. The SED and control microcosms were wrapped in foil to model the effects of sorption. While the PAR + SED and PAR + Effluent microcosms were irradiated under PAR lights, and modeled the cumulative effects of sorption and photodegradation. It was hypothesized that exposure to DBC sediment would decrease the number of CEC detected in the microcosms. Further, that the inclusion of PAR lights would produce an even greater decrease in CEC detection.

CEC detections were evaluated to answer three questions: (1) Were there CEC detected in the NWRF effluent? (2) Did DBC sediment decrease the number of

detections? and (3) Did the addition of PAR lights decrease the number of detections? Preliminary analysis answered yes to these questions. Over thirty CEC were present in all microcosms. Sediment and PAR interaction demonstrated the capacity to decrease CEC detects. However, PAR was more effective in reducing the total number of CEC detections, suggesting that photodegradation could be an important mechanism for CEC removal.

The same questions were answered for each class of CEC. Contact of the effluent with sediment appeared to reduce the number of pesticide detections. The effects of PAR were effective in reducing PPCP detects. EDCs, flame retardants, and preservatives were susceptible to the synergistic effects of sediment and PAR, and attenuation of artificial sweeteners and stimulants were negligible for both mechanisms. Observation of individual CEC class detection behavior point to the potential for Dave Blue to serve as an environmental buffer for CEC attenuation.

There were observed concentration decreases for 4-nonylphenol, triclosan, and lidocaine through incubation with sediment and PAR. However, the SED experiments produced greater changes in concentration for those compounds. Because the photodegradation half-life of 4-nonylphenol is 7.4 days (~ 178 hours), and the PAR + SED and PAR + Effluent microcosms received only 30 cumulative hours of irradiation the duration of the experiment may not have been sufficient to observe photodegradation of 4-nonylphenol. Sorption appeared to be a mechanism for the reduction of theobromine even though stimulant detections suggested that the effects of sediment incubation was negligible. Time was an important factor for

sucralose attenuation because of its persistence. Incubation in the SED, PAR + SED, and PAR + Effluent microcosms show that sorption and photodegradation have the potential to serve as pathways for sucralose attenuation, but these processes will require time.

5.1 Limitations

As a preliminary study, this research shows that Dave Blue Creek has the potential to decrease CEC, but more research is needed to understand individual CEC behavior in the natural environment. Several CEC investigated in the study were metabolites (e.g., cotinine, theobromine, 4-nonylphenol, and 1,7-dimethylxanthine) of compounds that were not as persistent (e.g., caffeine), or not investigated in the study because of their ability to degrade (e.g., nicotine). When CEC detections or concentrations decreased in the microcosms, it is unclear whether this is due to complete mineralization of the analyte or if the CEC produced metabolites that may be more persistent in the natural environment.

The CEC received 30 cumulative hours of simulated solar radiation, which may have resulted in a buffering effect (Cullin, 2014), meaning CEC concentrations were temporarily reduced during solar radiation. Yamamoto et al. (2009) studied the photolysis of CEC under direct sunlight and reported photodegradation half-lives of >50-hr for acetaminophen, carbamazepine, and ibuprofen. The two hours of simulated solar radiation in the PAR + SED and PAR + Effluent microcosms was estimated based on the canopy cover of DBC, and may not have been representative of the amount of time CEC would receive solar radiation in DBC.

During experimentation, CEC detections and concentrations show a relative decrease overtime. However, background sediment concentrations were not assessed in this research, meaning the effect of CEC desorption from sediment in the microcosms is unknown. Further, outside interferences from the laboratory and laboratory members could have increased analyte concentrations in the microcosms. Other laboratory members were present during effluent sampling and CEC extractions. Since CEC concentrations are sensitive to contamination use of albuterol, caffeine, sucralose, and other CEC by other laboratory members could have influenced analyte concentrations. Flame retardants (e.g., TCEP, TDCPP, and TCPP) have been detected at trace concentrations (ng/L) in indoor air, dust, and airborne particulate matter (Fan et al., 2014; Cheng et al., 2016), and could have contaminated the studies as well.

5.2 Future Work

Further research is recommended in the following areas:

- Sediment and PAR demonstrated the potential to reduce CEC concentrations. A few CEC investigated in the study were metabolites. More attention should be placed on how CEC degrades, what metabolites stem from their degradation, how they persistent in the environment, and their potential human health impact.
- Reaction kinetic data were lacking for many compounds in this research. More kinetic research should be performed to close this gap in knowledge. Increased

kinetic data would have aided in the understanding of CEC behavior in this research, and the natural environment.

- This research attempted to focus on sorption and photodegradation for possible attenuation, but CEC can also partition into fatty tissue of aquatic biota, volatilize, biodegrade, or be diluted (Kim and Tanaka, 2009). Currently, NWRF effluent is discharged into the Canadian River. Assessing how CEC concentrations change along different reaches of the Canadian River would provide a more accurate portrayal of what to expect from DBC as an environmental buffer.

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Appendix A. Water Quality Data

Table A-1 Nitrate-N concentrations measured before and after incubation in the microcosms.

Microcosm	Pre-Experiment (mg/L)	Post-Experiment (mg/L)
PAR + SED	12.9	7.11
PAR + SED	12.9	6.38
PAR + Effluent	12.9	11.5
SED	12.5	3.50
SED	12.5	4.14
Control	12.5	13.2

Table A-2 Nitrite-N concentrations measured before and after incubation in the microcosms.

Microcosm	Pre-Experiment (mg/L)	Post-Experiment (mg/L)
PAR + SED	0.112	<0.015
PAR + SED	0.112	<0.015
PAR + Effluent	0.112	0.466
SED	0.070	0.021
SED	0.070	0.031
Control	0.070	<0.015

Table A-3 Ammonia-N concentrations measured before and after incubation in the microcosms.

Microcosm	Pre-Experiment (mg/L)	Post-Experiment (mg/L)
PAR + SED	0.43	0.023
PAR + SED	0.43	0.021
PAR + Effluent	0.43	<0.015
SED	0.24	0.14
SED	0.24	0.12
Control	0.24	<0.015

Table A-4 Orthophosphorus concentrations measured before and after incubation in the microcosms.

Microcosm	Pre-Experiment (mg/L)	Post-Experiment (mg/L)
PAR + SED	1.86	<1.50
PAR + SED	1.86	<1.50
PAR + Effluent	1.86	1.83
SED	5.21	<1.50
SED	5.21	<1.50
Control	5.21	5.61

Table A-5 Soluble orthophosphorus concentrations measured before and after incubation in the microcosms.

Microcosm	Pre-Experiment (mg/L)	Post-Experiment (mg/L)
PAR + SED	1.74	<1.50
PAR + SED	1.74	<1.50
PAR + Effluent	1.74	1.79
SED	4.32	<1.50
SED	4.32	<1.50
Control	4.32	5.54

Table A-6 Effluent readings for 5-day BOD, dissolved oxygen, and pH in the microcosms prior to incubation.

Microcosm	5 - day BOD (mg/L)	Dissolved Oxygen (mg/L)	pH
PAR + SED	4.00	8.40	7.23
PAR + SED	4.00	8.40	7.23
PAR + Effluent	4.00	8.40	7.23
SED	3.90	5.80	7.15
SED	3.90	5.80	7.15
Control	3.90	5.80	7.15

Appendix B. CEC Concentration Data

Table B-1 Individual CEC concentration data for the SED microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	1500	1400	440	<100
4-tert-Octyphenol	120	240	<50	<50
Androstenedione	51	7.7	<5	<5
Bisphenol-A	<10	<10	<10	25
Estradiol	<5	<5	<5	200
Estriol	<5	<5	<5	<5
Estrone	<5	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	<5	<5	<5	<5
Progesterone	<5	<5	<5	<5
Testosterone	10	<5	6.4	<5
Acetaminophen	<5	<5	<5	18
Albuterol	<5	27	5.9	<5
Amoxicillin	970	<20	<20	<20
Atenolol	150	32	<5	<5
Azithromycin	<20	<20	<20	<20
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	<5	<5	<5
Carbamazepine	480	340	200	95
Carisoprodol	270	300	340	38
Chloramphenicol	<10	<10	<10	<10
Cimetidine	230	24	<5	<5
Dehydronifedipine	<5	<5	<5	<5
Diazepam	<5	<5	<5	<5
Diclofenac	<5	<5	<5	<5
Dilantin	<20	240	280	40
Diltiazem	<5	<5	<5	<5
Erythromycin	<10	<10	<10	<10
Flumequine	<10	<10	<10	<10
Fluoxetine	<10	<10	<10	<10
Gemfibrozil	67	<5	<5	<5
Ibuprofen	<10	<10	<10	<10

Table B-1 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	480	190	78	<10
Iopromide	<5	<5	<5	<5
Ketoprofen	<5	<5	5.1	<5
Ketorolac	<5	<5	22	5.5
Lidocaine	940	610	310	80
Lincomycin	<10	<10	<10	<10
Lopressor	120	<20	33	<20
Meclofenamic Acid	<5	<5	17	<5
Meprobamate	<5	<5	500	260
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	<5	<5
Primidone	<5	<5	70	65
Salicylic Acid	<100	<100	<100	<100
Sulfachloropyridazine	<5	<5	<5	<5
Sulfadiazine	<5	<5	<5	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfamethoxazole	5200	6700	1500	730
Sulfathiazole	<5	<5	<5	<5
Theophylline	<5	<5	100	<5
Warfarin	<20	<20	<20	<20
1,7- Dimethylxanthine	<10	<10	<10	<10
Caffeine	45	<5	8.6	13
Cotinine	<10	<10	<10	<10
Theobromine	380	180	59	22
Butylparaben	<10	<10	<10	<10
Ethylparaben	<5	<5	<5	<5
Isobutylparaben	<20	<20	<20	<20
Methylparaben	<5	<5	<5	<5

Table B-1 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<20	<20	<20	<20
Thiabendazole	73	<5	<5	<5
Triclocarban	<5	<5	<5	<5
Triclosan	<5	<5	<5	<5
Trimethoprim	850	150	35	<10
Acesulfame-K	6600	1200	660	400
Sucralose	180000	200000	57000	56000
2,4- D	<100	<100	<100	<100
Atrazine	<5	<5	6.8	<5
Bromacil	15	<5	7.8	<5
Chloidazon	15	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	17	7.5	26	7.5
Cyanizine	85	<5	27	<5
DACT	220	660	92	22
DEA	<5	<5	11	<5
DEET	62	<5	90	89
DIA	340	600	170	81
Diuron	8.6	<5	<5	<5
Isoproturon	<5	<5	<5	<5
Linuron	<100	<100	<100	<100
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	<5	<5	64	<5
Simazine	2200	2500	1100	620
TCEP	580	890	900	<10
TCPP	390	370	<100	<100
TDCPP	260	180	<100	120

Table B-2 Individual CEC concentration data for the SED duplicate microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	1500	470	680	<100
4-tert-Octyphenol	120	68	<50	<50
Androstenedione	51	7.6	<5	<5
Bisphenol-A	<10	96	<10	<10
Estradiol	<5	<5	<5	<5
Estriol	<5	<5	<5	<5
Estrone	<5	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	<5	<5	<5	<5
Progesterone	<5	<5	<5	<5
Testosterone	10	<5	9	<5
Acetaminophen	<5	<5	<5	27
Albuterol	<5	8.5	<5	<5
Amoxicillin	970	1100	20	<20
Atenolol	150	<5	<5	<5
Azithromycin	<20	<20	<20	<20
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	<5	<5	<5
Carbamazepine	480	160	130	83
Carisoprodol	270	140	220	43
Chloramphenicol	<10	<10	<10	<10
Cimetidine	230	8.3	<5	<5
Dehydronifedipine	<5	<5	<5	<5
Diazepam	<5	<5	<5	<5
Diclofenac	<5	<5	<5	<5
Dilantin	<20	63	470	45
Diltiazem	<5	<5	<5	<5
Erythromycin	<10	<10	<10	<10
Flumequine	<10	<10	12	<10
Fluoxetine	<10	<10	<10	<10
Gemfibrozil	67	<5	7	<5
Ibuprofen	<10	<10	<10	<10

Table B-2 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	480	180	50	<10
Iopromide	<5	<5	<5	<5
Ketoprofen	<5	<5	9.6	<5
Ketorolac	<5	<5	22	<5
Lidocaine	940	330	210	130
Lincomycin	<10	<10	<10	<10
Lopressor	120	32	<20	<20
Meclofenamic Acid	<5	<5	19	<5
Meprobamate	<5	<5	470	270
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	<5	<5
Primidone	<5	<5	41	70
Salicylic Acid	<100	<100	<100	<100
Sulfachloropyridazine	<100	<100	<100	<100
Sulfadiazine	<5	<5	<5	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfamethoxazole	5200	970	1300	590
Sulfathiazole	<5	<5	<5	<5
Theophylline	<5	<5	43	38
Warfarin	<20	<20	5.7	5
1,7- Dimethylxanthine	<10	<10	<10	<10
Caffeine	45	<5	34	6.1
Cotinine	<10	<10	120	<10
Theobromine	380	81	50	13
Butylparaben	<10	<10	<10	<10
Ethylparaben	<5	<5	<5	<5
Isobutylparaben	<20	<20	<20	<20
Methylparaben	<5	<5	<5	<5

Table B-2 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<20	<20	<20	<20
Thiabendazole	73	<5	<5	<5
Triclocarban	<5	<5	<5	<5
Triclosan	<5	<5	<5	<5
Trimethoprim	850	26	9.7	<10
Acesulfame-K	6600	350	590	380
Sucralose	180000	49000	61000	57000
2,4- D	<100	<100	<100	<100
Atrazine	<5	<5	5.1	<5
Bromacil	15	<5	6.5	<5
Chloidazon	15	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	17	7.5	<5	<5
Cyanizine	85	<5	20	<5
DACT	220	40	82	15
DEA	<5	<5	13	<5
DEET	62	76	92	14
DIA	340	140	120	72
Diuron	8.6	<5	<5	<5
Isoproturon	<5	<5	<5	<5
Linuron	<100	<100	<100	<100
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	<5	<5	56	<5
Simazine	2200	820	1000	570
TCEP	580	430	1100	<10
TCPP	390	500	<100	<100
TDCPP	260	190	120	<100

Table B-3 Individual CEC concentration data for the PAR + SED microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	280	1600	1500	1200
4-tert-Octyphenol	<50	<50	<50	<50
Androstenedione	<5	<5	<5	<5
Bisphenol-A	<10	<10	22	<10
Estradiol	<5	<5	<5	<5
Estriol	<5	<5	<5	<5
Estrone	15	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	20	<5	8.7	<5
Progesterone	<5	<5	<5	<5
Testosterone	11	6.9	8.2	<5
Acetaminophen	34	<5	<5	<5
Albuterol	<5	<5	6.9	<5
Amoxicillin	180	1400	460	<20
Atenolol	340	10	<5	<5
Azithromycin	<10	<10	<10	<10
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	<5	<5	<5
Carbamazepine	110	170	130	68
Carisoprodol	13	<5	410	50
Chloramphenicol	<10	<10	<10	<10
Cimetidine	690	<5	13	<5
Dehydronifedipine	5.8	<5	<5	<5
Diazepam	<5	<5	<5	<5
Diclofenac	66	<5	<5	<5
Dilantin	49	2100	320	<20
Diltiazem	<5	<5	<5	<5
Erythromycin	<10	<10	<10	<10
Flumequine	<10	140	85	<10
Fluoxetine	<10	<10	<10	<10
Gemfibrozil	39	<5	<5	<5
Ibuprofen	520	<10	<10	<10

Table B-3 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	1100	220	110	220
Iopromide	5.4	<5	<5	<5
Ketoprofen	<5	18	37	<5
Ketorolac	36	22	25	<5
Lidocaine	1000	820	690	43
Lincomycin	<10	<10	<10	<10
Lopressor	100	56	24	<20
Meclofenamic Acid	67	110	60	<5
Meprobamate	190	500	1000	<5
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	23	<5
Primidone	75	64	120	<5
Salicylic Acid	580	<100	<100	<100
Sulfachloropyridazine	<5	<5	<5	<5
Sulfadiazine	<5	<5	<5	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfamethoxazole	<5	3100	1400	<5
Sulfathiazole	<5	<5	<5	<5
Theophylline	<20	100	<20	30
Warfarin	5.4	<5	<5	<5
1,7- Dimethylxanthine	12	14	<10	<10
Caffeine	<5	<5	26	7
Cotinine	<10	110	150	<10
Theobromine	42	29	52	38
Butylparaben	<5	<5	<5	28
Ethylparaben	<20	<20	<20	<20
Isobutylparaben	<5	<5	<5	<5
Methylparaben	<20	<20	<20	<20

Table B-3 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<5	<5	<5	<5
Thiabendazole	<5	<5	<5	<5
Triclocarban	<5	<5	<5	<5
Triclosan	22	<10	<10	<10
Trimethoprim	440	130	33	<5
Acesulfame-K	140	260	<20	160
Sucralose	42000	60000	55000	3800
2,4- D	62	12	<5	<5
Atrazine	<5	5.6	<5	<5
Bromacil	<5	<5	6.3	<5
Chloidazon	<5	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	<5	<5	<5	<5
Cyanizine	<5	<5	<5	9.8
DACT	13	<5	44	<5
DEA	<5	11	<5	5
DEET	<10	100	160	16
DIA	46	41	87	45
Diuron	7.5	5.7	<5	<5
Isoproturon	<100	<100	<100	<100
Linuron	<5	<5	<5	<5
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	8.5	150	32	<5
Simazine	300	430	360	<5
TCEP	210	830	1100	840
TCPP	560	<100	<100	<100
TDCPP	370	140	<100	<100

Table B-4 Individual CEC concentration data for the PAR + SED duplicate microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	280	970	2800	240
4-tert-Octyphenol	<50	<50	<50	<50
Androstenedione	<5	<5	<5	<5
Bisphenol-A	<10	<10	<10	<10
Estradiol	<5	<5	<5	<5
Estriol	<5	<5	<5	<5
Estrone	15	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	20	<5	<5	<5
Progesterone	<5	<5	<5	<5
Testosterone	11	6.6	12	<5
Acetaminophen	34	<5	<5	<5
Albuterol	<5	<5	<5	<5
Amoxicillin	180	710	760	<20
Atenolol	340	14	50	<5
Azithromycin	<10	<10	<10	<10
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	<5	91	<5
Carbamazepine	110	130	270	61
Carisoprodol	13	480	250	69
Chloramphenicol	<10	<10	<10	<10
Cimetidine	690	8.4	<5	<5
Dehydronifedipine	5.8	<5	11	<5
Diazepam	<5	<5	6.2	<5
Diclofenac	66	<5	<5	<5
Dilantin	49	210	690	<20
Diltiazem	<5	11	100	<5
Erythromycin	<10	<10	11	<10
Flumequine	<10	120	830	<10
Fluoxetine	<10	<10	56	<10
Gemfibrozil	39	<5	54	11
Ibuprofen	520	<10	<10	<10

Table B-4 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	1100	230	350	90
Iopromide	5.4	<5	<5	<5
Ketoprofen	<5	7.4	<5	<5
Ketorolac	36	16	32	<5
Lidocaine	1000	550	1700	45
Lincomycin	<10	<10	<10	<10
Lopressor	100	85	210	<20
Meclofenamic Acid	67	90	140	<5
Meprobamate	190	590	1100	160
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	<5	<5
Primidone	75	61	200	7.9
Salicylic Acid	580	<100	<100	<100
Sulfachloropyridazine	<5	<5	<5	<5
Sulfadiazine	<5	<5	8.6	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfamethoxazole	<5	1100	1900	<5
Sulfathiazole	<5	<5	<5	<5
Theophylline	<20	<20	<20	<20
Warfarin	5.4	<5	<5	<5
1,7- Dimethylxanthine	12	<10	40	<10
Caffeine	<5	<5	45	<5
Cotinine	<10	80	56	<10
Theobromine	42	52	52	33
Butylparaben	<5	<5	<5	<5
Ethylparaben	<20	<20	<20	<20
Isobutylparaben	<5	<5	<5	<5
Methylparaben	<20	<20	<20	<20

Table B-4 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<5	<5	<5	<5
Thiabendazole	<5	<5	6	<5
Triclocarban	<5	<5	<5	<5
Triclosan	22	<10	<10	<10
Trimethoprim	440	130	740	<5
Acesulfame-K	140	240	280	62
Sucralose	42000	47000	54000	20000
2,4- D	62	37	130	<5
Atrazine	<5	<5	<5	<5
Bromacil	<5	6.7	11	<5
Chloidazon	<5	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	<5	<5	<5	<5
Cyanizine	<5	<5	<5	9.1
DACT	13	54	75	16
DEA	<5	<5	<5	5
DEET	<5	240	48	17
DIA	46	110	68	48
Diuron	7.5	<5	12	<5
Isoproturon	<100	<100	<100	<100
Linuron	<5	<5	<5	<5
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	8.5	110	69	<5
Simazine	300	430	520	<5
TCEP	210	620	450	780
TCPP	560	<100	1600	<100
TDCPP	370	140	330	<100

Table B-5 Individual CEC concentration data for the PAR + Effluent microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	280	1500	1600	240
4-tert-Octyphenol	<50	<50	<50	<50
Androstenedione	<5	6.1	<5	<5
Bisphenol-A	<10	<10	<10	<10
Estradiol	<5	<5	<5	<5
Estriol	<5	<5	<5	<5
Estrone	15	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	20	<5	<5	<5
Progesterone	<5	<5	<5	<5
Testosterone	11	11	8.7	<5
Acetaminophen	34	<5	<5	<5
Albuterol	<5	<5	<5	<5
Amoxicillin	180	760	370	640
Atenolol	340	57	<5	17
Azithromycin	<10	<10	<10	<10
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	230	<5	<5
Carbamazepine	110	240	160	150
Carisoprodol	13	560	490	33
Chloramphenicol	<10	<10	<10	<10
Cimetidine	690	<5	<5	<5
Dehydronifedipine	5.8	5.6	<5	<5
Diazepam	<5	6.1	<5	<5
Diclofenac	66	<5	<5	<5
Dilantin	49	1400	450	120
Diltiazem	<5	120	<5	<5
Erythromycin	<10	18	<10	<10
Flumequine	<10	360	22	<10
Fluoxetine	<10	75	<10	<10
Gemfibrozil	39	<5	42	19
Ibuprofen	520	<10	<10	<10

Table B-5 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	1100	360	140	1100
Iopromide	5.4	<5	<5	<5
Ketoprofen	<5	<5	35	<5
Ketorolac	36	19	20	<5
Lidocaine	1000	600	640	92
Lincomycin	<10	<10	<10	<10
Lopressor	100	220	36	380
Meclofenamic Acid	67	130	32	150
Meprobamate	190	620	1000	150
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	<5	<5
Primidone	75	57	100	10
Salicylic Acid	580	<100	<100	100
Sulfachloropyridazine	<5	<5	<5	<5
Sulfadiazine	<5	11	6	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfamethoxazole	<5	1300	1100	<5
Sulfathiazole	<5	<5	<5	<5
Theophylline	<20	<20	<20	<20
Warfarin	5.4	<5	<5	<5
1,7- Dimethylxanthine	12	12	<10	<10
Caffeine	<5	7.4	65	<5
Cotinine	<10	30	130	<10
Theobromine	42	52	58	38
Butylparaben	<5	<5	<5	<5
Ethylparaben	<20	<20	<20	<20
Isobutylparaben	<5	<5	<5	<5
Methylparaben	<20	<20	<20	<20

Table B-5 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<5	<5	<5	<5
Thiabendazole	<5	13	<5	<5
Triclocarban	<5	<5	<5	<5
Triclosan	22	11	<10	<10
Trimethoprim	440	900	45	510
Acesulfame-K	140	250	<20	54
Sucralose	42000	61000	56000	15000
2,4- D	62	120	<5	<5
Atrazine	<5	6.2	<5	6
Bromacil	<5	12	<5	<5
Chloidazon	<5	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	<5	<5	5.5	<5
Cyanizine	<5	5.2	<5	14
DACT	13	79	63	15
DEA	<5	9.2	<5	7.8
DEET	<10	50	120	15
DIA	46	73	64	65
Diuron	7.5	14	<5	49
Isoproturon	<100	<100	<100	<100
Linuron	<5	<5	<5	<5
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	8.5	75	57	14
Simazine	300	550	330	<5
TCEP	210	440	1000	310
TCPP	560	1700	<100	720
TDCPP	370	310	120	240

Table B-6 Individual CEC concentration data for the control microcosm over time.

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
4-nonylphenol	1500	2700	2000	<100
4-tert-Octyphenol	120	96	<50	<50
Androstenedione	51	9	<5	<5
Bisphenol-A	<10	<10	<10	<10
Estradiol	<5	<5	<5	<5
Estriol	<5	<5	<5	<5
Estrone	<5	<5	<5	<5
Ethinyl Estradiol- 17α	<5	<5	<5	<5
Norethisterone	<5	<5	<5	<5
Progesterone	<5	6.4	<5	<5
Testosterone	10	<5	10	<5
Acetaminophen	<5	<5	<5	34
Albuterol	<5	47	<5	50
Amoxicillin	970	940	330	<20
Atenolol	150	130	43	110
Azithromycin	<20	<20	<20	<20
Bendroflumethiazide	<5	<5	<5	<5
Bezafibrate	<5	<5	<5	<5
Butalbital	<5	<5	<5	<5
Carbadox	<5	<5	<5	<5
Carbamazepine	480	420	320	160
Carisoprodol	270	300	320	33
Chloramphenicol	<10	<10	<10	<10
Cimetidine	230	210	7.1	<5
Dehydronifedipine	<5	<5	6	<5
Diazepam	<5	<5	<5	<5
Diclofenac	<5	<5	<5	45
Dilantin	<20	180	1100	54
Diltiazem	<5	<5	72	16
Erythromycin	<10	<10	15	<10
Flumequine	<10	<10	82	<10
Fluoxetine	<10	<10	94	<10
Gemfibrozil	67	<5	15	10
Ibuprofen	<10	<10	<10	<10

Table B-6 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Iohexal	480	300	400	890
Iopromide	<5	<5	17	34
Ketoprofen	<5	<5	<5	<5
Ketorolac	<5	<5	36	<5
Lidocaine	940	1300	640	340
Lincomycin	<10	<10	<10	<10
Lopressor	120	<20	200	72
Meclofenamic Acid	<5	<5	130	47
Meprobamate	<5	<5	860	250
Naproxen	<10	<10	<10	<10
Nifedipine	<20	<20	<20	<20
Oxolinic Acid	<10	<10	<10	<10
Pentoxifylline	<5	<5	<5	<5
Phenazone	<5	<5	<5	<5
Primidone	<5	<5	72	60
Salicylic Acid	<100	<100	<100	<100
Sulfachloropyridazine	<100	<100	<100	<100
Sulfadiazine	<5	<5	6	<5
Sulfadimethoxine	<5	<5	<5	<5
Sulfamazerine	<5	<5	<5	<5
Sulfamethazine	<5	<5	<5	<5
Sulfamethizole	<5	<5	<5	<5
Sulfmethoxazole	5200	7100	1800	<5
Sulfathiazole	<5	<5	<5	<5
Theophylline	<5	<5	<5	<5
Warfarin	<20	<20	<20	<20
1,7- Dimethylxanthine	<10	<10	<10	<10
Caffeine	45	<5	8.9	5
Cotinine	<10	<10	33	<10
Theobromine	380	160	52	49
Butylparaben	<10	<10	<10	<10
Ethylparaben	<5	<5	<5	<5
Isobutylparaben	<20	<20	<20	<20
Methylparaben	<5	<5	<5	<5

Table B-6 (Continued).

Compound	Initial (ng/L)	Day 5 (ng/L)	Day 10 (ng/L)	Day 15 (ng/L)
Propylparaben	<20	<20	<20	<20
Thiabendazole	73	39	5	<5
Triclocarban	<5	<5	<5	<5
Triclosan	<5	<5	<5	<5
Trimethoprim	850	39	520	280
Acesulfame-K	6600	1100	580	320
Sucralose	180000	240000	59000	52000
2,4- D	<5	<5	43	<5
Atrazine	<5	<5	<5	<5
Bromacil	15	14	10	<5
Chloidazon	15	<5	<5	<5
Chlorotoluron	<5	<5	<5	<5
Clofibric Acid	17	<5	50	<5
Cyanizine	85	<5	<5	<5
DACT	220	430	150	30
DEA	<5	<5	<5	5.2
DEET	62	60	30	11
DIA	340	470	230	100
Diuron	8.6	6.4	9.9	5.4
Isoproturon	<5	<5	<5	<5
Linuron	<100	<100	<100	<100
Metazachlor	<5	<5	<5	<5
Metolachlor	<5	<5	<5	<5
OUST	<5	<5	<5	<5
Propazine	<5	<5	<5	<5
Quinoline	<5	<5	49	9.3
Simazine	2200	3100	1400	1200
TCEP	580	<10	360	210
TCPP	390	220	1200	540
TDCPP	260	230	310	310