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The Effects of Stormwater on Wastewater Treatability of Targeted Emerging Contaminants at a Wastewater Treatment Plant

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Municipal wastewater treatment plants have traditionally been designed to treat conventional pollutants found in sanitary wastewaters. However, many synthetic pollutants, such as pharmaceuticals and personal care products, also enter the wastewater stream. Some of these nontraditional contaminants are not efficiently removed by the treatment process at the wastewater treatment plant. These chemicals, commonly referred to as emerging contaminants, have been identified in surface waters receiving wastewater effluents and have been found to potentially cause adverse effects on aquatic wildlife. These materials are produced in very large quantities and are disposed of in sewage where partial treatment occurs before their discharge. Some of the pharmaceuticals excreted from the human user's body are metabolized and are more toxic and untreatable than their parent compound.

Factors that can influence their treatability by wastewater treatment systems include their physical and chemical characteristics, the retention time in the unit treatment processes, and flow rates that can be influenced by rainfall in the sanitary wastewater service area. In our EPA funded research, we are examining the effect of stormwater infiltration and inflow into sanitary systems on the amounts and treatability of targeted pharmaceuticals, PAHs and pesticides at the treatment plants. Seven dry weather and seven wet weather series of samples have been obtained from several locations of the treatment plant and are being compared to determine if

there are significant changes in influent quantities and removals during wet weather. From preliminary data, treatability appears to remain similar during both wet and dry weather conditions. Hydraulic retention times and hourly flow variations are being examined during the final portion of this project to identify further effects.

11.1 Background

11.1.1 History

The U.S. Environmental Protection Agency (EPA) sets guidelines for pollutant discharges by using regulations contained in the National Pollutant Discharge Elimination System (NPDES). This regulation monitors point sources such as municipal and industrial wastewater treatment systems. These regulations mainly focus on conventional pollutant discharges. However, new classes of unregulated contaminants have become an emerging environmental problem (Petrovic, Gonzalez, and Barceló 2003, 685-696). These pollutants have recently been found in waterways and in groundwater. Pharmaceuticals were first reported in surface waters during the investigation of U.S. waterways in the 1970s, although they are not regulated as legacy pollutants such as PCBs and DDTs (Snyder et al. 2006). Researchers such as Watts et al (1983) first reported the occurrence of several selected antibiotics in river water samples; since then there have been many investigations of antibiotics and publications documenting their presence in groundwaters, surface waters, wastewaters and landfill leachates (Xu et al. 2007, 4526-4534). The EPA is working in conjunction with the US Geological Survey to compile a list of these contaminants that are in the US waterways (*A National Reconnaissance*). Samples have been obtained from 139 US streams and waterways to analyze 95 organic wastewater contaminants (Kolpin et al. 2002, 1202-1211). These emerging contaminants are being used in large quantities and in everyday use and many have no maximum concentration limits in discharge permits. Research on several contaminants that were found in the *Reconnaissance* study is being conducted to de-

termine the potential effect of these compounds on aquatic wildlife and the environment. As an example, Campbell (2006) conducted a study to determine the effect of estrogen, an endocrine chemical disruptor on aquatic wildlife.

11.1.2 Importance

Emerging contaminants as defined by the U.S. Geological Survey are “any synthetic or naturally occurring chemical or any microorganism that is not commonly monitored in the environment but has the potential to enter the environment and cause known or suspected adverse ecological and (or) human health effects.” Little is known about the effects of these compounds in the environment. There have been studies on the effects of some of these pollutants on wildlife. Endocrine disrupting chemicals, a sub-category of emerging contaminants, have caused sexual abnormalities in certain species of fish. Endocrine disrupting chemicals include a broad range of chemicals: natural and synthetic estrogens, pesticides, and industrial chemicals (Campbell et al. 2006, 1265-1280). Low levels (ng/L) of waterborne estrogens lead to adverse effects such as the feminization of fish, impaired reproduction and abnormal sexual development (Sellin et al. 2009, 14-21).

Research on emerging contaminants has been improved with new analytical methods that can quantify emerging contaminants in trace quantities. Some emerging contaminants could possibly cause adverse effects on the ecosystem even in small quantities. Studies are being done to determine the fate and transport of these chemicals from their point sources (or non-point) sources to the environment and to reduce the emission concentrations. The disposal of unused medication via the toilet seems to be of minor importance but many of the pharmaceuticals applied in human medical care are not completely eliminated in the human body. Often they are excreted only slightly transformed or even unchanged mostly conjugated to polar molecules (e.g. as glucoronides) (Heberer 2002, 5-17).These conjugates can pass through the treatment plant untreated and enter into the waterways. Some residue of

contaminants may also leach into groundwater aquifers and have already been reported to occur in ground and drinking water samples from water works using bank filtration or artificial groundwater recharge downstream from municipal STPs (Heberer 2002, 5-17).

An important point source of emerging contaminants is municipal conventional wastewater treatment systems. Emerging contaminants can be divided into different categories: pharmaceuticals and personal care products (PPCPs); polycyclic aromatic hydrocarbons (PAHs); pesticides, heavy metals and microbes. Pharmaceuticals enter the treatment system either directly, or through fecal matter or urine. Personal care products could possibly enter the treatment plant through direct disposal or by shower or bath water. Pesticides, PAHs, heavy metals, and microbial material can enter the treatment plant through urban runoff that infiltrates the sewer lines. Some emerging contaminants may not be adequately removed by wastewater treatment facilities. Recent studies have shown that wastewater treatment plant removal of personal care products and pharmaceutical can range between 60% and 90% for a variety of polar compounds (Carballa et al. 2004, 2918-2926). The removal rate is mostly contingent on the physical and chemical nature of the pollutant and the effect of the wastewater matrix. It also depends on the treatment plant itself: the retention time through each unit process, and the specific unit processes used at the treatment facility. During our study, we are examining the effects of increases in flow rates and changes in influent concentrations during significant rain events on treatability of these compounds.

11.1.3 Pollutants and their characteristics

Emerging contaminants include pharmaceuticals and personal care products, PAHs, pesticides, heavy metals and microbes. For our study, we are focusing on pharmaceuticals, PAHs and pesticides, as listed in Table 1, although parallel investigations are examining the sources and fates of urban area microorganisms. In order to es-

timate how and why these pollutants are affected by the treatment plant, the physical and chemical characteristics have to be known.

Table 1. Pharmaceuticals being investigated

Chemical Name	**Molecular Weight g/mol**	**Log k_{ow}**	**Solubility(mg/L)**
Carbamazepine	236.1	2.45	17.7
Fluoxetine	309.3	4.05	38.4
Gemfibrozil	250.12	4.78	5.0
Ibuprofen	206	3.5-4.0	41.5
Sulfamethoxazole	253	0.9	600
Triclosan	289.5	4.8-5.4	2-4.6
Trimethoprim	290.32	0.79	400

(Goodson et al. 2012)

Table 2. PAHs being investigated

Chemical Name	**Molecular Weight (g/mol)**	**Solubility (mg/L)**	**Log k_{ow}**
naphthalene	128.2	31.5	3.30
acenaphthylene	152.2	3.93	4.07
acenaphthene	154.2	16.1	3.94
fluorene	166.2	1.98	4.23
anthracene	178.2	0.07	4.54
phenanthrene	178.2	1.12	4.57
pyrene	202.2	0.135	5.18
fluoranthene	202.2	0.264	5.14
benzo[a]anthracene	228.3	0.014	5.66
chrysene	228.3	1.8×10^{-3}	5.71

(Goodson et al. 2012)

Table 3. Pesticides being investigated

Chemical Name	**Molecular Weight (g/mol)**	**Solubility (mg/L)**	**Log k_{ow}**
Methoxychlor	345.65	0.1	4.68-5.08
Aldrin	364.91	0.027	6.5
Dieldrin	380.91	0.1	6.2
Chlordane	409.76	insoluble*	~5.54
Arochlor Σ	257.9-453	insoluble*	5.6-6.8
Lindane	290.83	17	3.8
Heptachlor	373.32	0.056	6.10
Heptachlor-epoxide	389.40	not found	5.40

(Goodson et al. 2012)

Pharmaceuticals have a variety of chemical properties. The properties of these chemicals that affect their mobility and treatment in wastewater treatment include: solubility, sorption to suspended solids and particulates, and how it reacts for different pH conditions of the solution. Chemicals with relatively high solubility and low sorption would be harder to eliminate in wastewater than a chemical with high sorption and lower solubility. The ability to sorb onto particulates is measured by the log of the octanol-water coefficient (log k_{ow}).

As an example, carbamazepine has been difficult to remove from wastewater as it is very soluble in water (with a solubility of 17.7 mg/L) and has a relatively low sorption rate (log k_{ow} of 2.45). Many studies on removal efficiencies of carbamazepine show that carbamazepine is difficult to removal from sewage (Nakada et al. 2006, 3297-3303); (Sethia and Squillante 2004, 1-10); (Zhang, Geißen, and Gal 2008, 1151-1161). Due to it persistent nature, carbamazepine has been proposed as a molecular marker of sewage (Nakada et al. 2006, 3297-3303). At low concentrations, carbamazepine is also resistant to biodegradation (Zhang, Geißen, and Gal 2008, 1151-1161). Carbamazepine has been frequently detected in groundwater up to concentrations of 610 ng/L and in other water bodies (Zhang, Geißen, and Gal 2008, 1151-1161).

Sulfamethoxazole is soluble in water (solubility of 600 mg/L) and has a low sorption rate (log k_{ow} of 0.79). At pH levels above 5.7, sulfamethoxazole remains as an anionic species, neutral at pH values between 1.7 and 5.7, and positive at pH levels below 1.7 (Nghiem, Schäfer, and Elimelech 2005, 7698-7705). Ibuprofen, triclosan, gemfibrozil and fluoxetine have high kow coefficients that (ranging from 3.5 to 5.4) under neutral pH conditions. Under neutral conditions they theoretically would sorb to particulates, but could be ionized if there was a reduction in pH.

PAHs are highly lipophilic so they do not readily dissolve in aqueous solutions. They are divided into two categories: high molecular weight PAHs and low molecular weight PAHs. Naphthalene is a LMW PAH and it maybe be slightly more soluble that the other PAHs with higher molecular weights. PAHs are molecular compounds so they have no ionization potential in aqueous solutions. PAHs tend to sorb onto particulate matter and theoretically should be removed during basic sedimentation processes, such as during primary sedimentation.

Katsoyiannis et al (2004) categorizes pesticides as persistent organic pollutants. These compounds tend to have low water and high fat solubility, stability during degradation processes, low vapor pressure, and are persistent in the environment (Katsoyiannis and Samara 2004, 2685-2698)(Monteith et al. 1995, pp. 964-970). Pesticides can enter wastewater treatment plants by surface runoff from treated sites, by contaminated rinses from cleaning of pesticide applicators and containers, and/or from disposal of unused pesticides (Monteith et al. 1995, pp. 964-970). If pesticides are transported by urban runoff, they can enter the groundwater or sanitary sewers. The removal of pesticides from water is one of the major environmental concerns today (AHMAD et al. 2010, 231-271).

11.2 Background on Project

11.2.1 Description of Site

Tuscaloosa is the fifth largest city in the state of Alabama with a population of about 83,000, according to the 2006 Census. The total area of Tuscaloosa is 66.7 mi^2, with 10.5 mi^2 comprised of surface water (Lake Tuscaloosa and the Black Warrior River) (Wikipedia-Tuscaloosa)(City of Tuscaloosa and Wastewater Management). Typical weather is humid with total annual rainfalls being about 60 inches. Tuscaloosa wastewater treatment occurs at the Earl Hilliard Wastewater Treatment Plant.

Earl Hilliard Wastewater Treatment Plant

Tuscaloosa's wastewater treatment system consists of approximately 550 miles of City maintained collection lines with another 50 miles of privately owned collection lines. Over 60 sanitary sewer lift stations are also used in the city. Tuscaloosa's wastewater facility was built in 1960 and upgraded in 1974 (City of Tuscaloosa and Wastewater Management). A $33 million expansion was designed in 1995, increasing the capacity of the treatment plant to 24 million gallons per day (City of Tuscaloosa and Wastewater Management). The Earl Hilliard Wastewater facility has primary and secondary treatment components that are duplicat-

ed in case part of the plant needs to shut down for maintenance or any technical failure.

Most wastewater treatment plants in the US, such as the Earl Hilliard facility, are typically secondary treatment systems with a pretreatment phase, a primary clarifier, an aeration tank, a secondary clarifier and disinfection system. Tuscaloosa's wastewater treatment uses ultraviolet disinfection instead of chlorine. An anaerobic digester is used for treatment of the sludge. Frequent monitoring of performance focusses on conventional pollutants (BOD_5, $CBOD_5$, NH_3-N, TKN, pH, and TSS).

11.2.2 Methodology

11.2.2.1 Sampling method

During each sampled event, one liter composite samples of wastewater were manually collected from four locations at the wastewater treatment facility: the inlet of the treatment plant before pre-treatment, the effluent from the primary clarifier, the effluent from the secondary clarifier and at the final discharge outlet, after disinfection. Each sample was a composite sample taken during a two hour time period. Six one liter sample bottles were used at each sampling location for the analysis of acidic and basic pharmaceuticals, PAHs, and pesticides. Each sample was stored in amber glass bottles, and refrigerated before extraction.

11.2.2.2 Analysis

The acidic pharmaceuticals were extracted using SPE extraction (EPA method 1694). Samples for acidic pharmaceutical analysis were acidified to 2.0 ± 0.1 pH using hydrochloric acid. Sodium ethylenediaminetetraacetic acid (Na-EDTA) was added for chelation of any heavy metals that might be present. SPE cartridges

were eluted with methanol and 2.0 pH reagent water before extraction.

Samples for basic pharmaceutical analyses were adjusted to a pH of 10 ± 0.1 using ammonium hydroxide. SPE cartridges for basic analyses were eluted using methanol and dionized water. Polycyclic aromatic hydrocarbons (PAHs) were extracted using separation funnels with kuderna danish (KD) concentration. Samples for pesticides were sent to Penn State Harrisburg for analyses, also after separation funnel and KD sample preparation. The analyses were conducted using HPLC for the ECs, GC-ECD for the pesticides and GC-MS for the PAHs.

11.2 Results and Discussions

11.2.1 Results

We compared the treatment plant samples taken during normal weather conditions with samples taken during rain events at each of the four locations. Seven sample sets taken during wet weather were compared to seven sample sets taken during dry weather. Currently, we are still in the process of completing the analyses. Figures A and B are examples of some PAHs during a wet weather and a dry weather event.

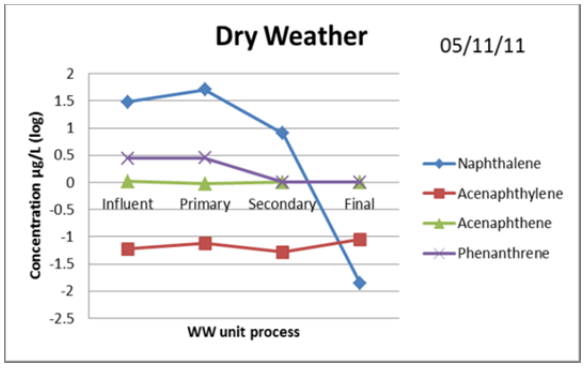


Figure A. Dry Weather data for PAHs

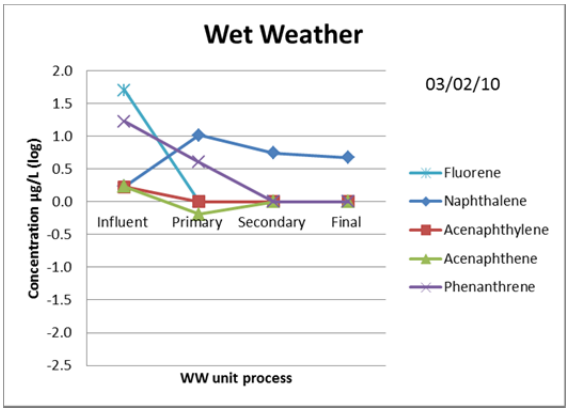
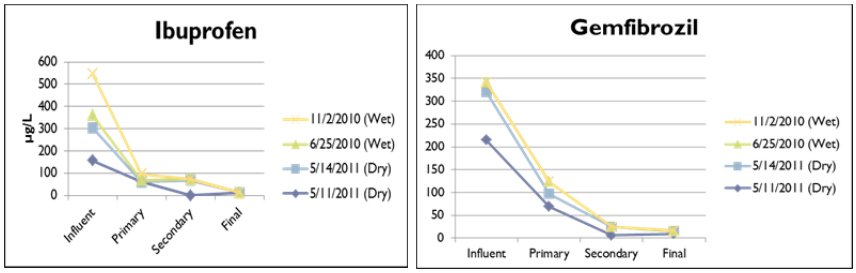


Figure B. Wet weather data for PAHs

As shown on Figures A and B, the concentrations are being compared using log-transformed values to better cover the ranges of the observed concentrations. For the PAH analyses, there is a steady reduction in concentration for the dry weather for naphthalene and phenanthrene from the influent to the final effluent. Acenaphthene and acenaphthylene do not show significant removals during dry weather conditions. During wet weather, naphthalene shows an increase during the primary phase before decreasing during secondary treatment in contrast to its treatment during dry weather. Phenanthrene shows a steady decrease in treatment during both wet and dry weather. Acenaphthylene and acenaphthene both show decreases during primary treatment. Acenaphthene appears to slightly increase during secondary treatment. During dry weather, the greatest treatability for most compounds studied occurred during the secondary treatment phase. Acenaphthene showed no treatability during dry weather, while it showed a small decrease during wet weather. For the wet weather samples, acenaphthylene, acenaphthene and fluorene all showed decreases in concentrations during the primary treatment phase.

In this paper, we are comparing dry weather and wet weather samples for two pharmaceuticals: ibuprofen and gemfibrozil. Data for the other pharmaceuticals are still being analyzed. Ibuprofen and gemfibrozil both showed a steady decrease in concentration during

both wet weather and dry weather conditions. Both compounds also showed a significant reduction in the primary treatment process for both weather conditions.



Figures C and D. Comparison of wet and dry weather concentrations

Each wet weather sample set was taken during a rain event. For each rain event, the measured rainfall was documented and compared to the flow rate of the treatment plant for that day. The concentration for each sample set was compared to the daily flow rate and the rainfall data accumulated.

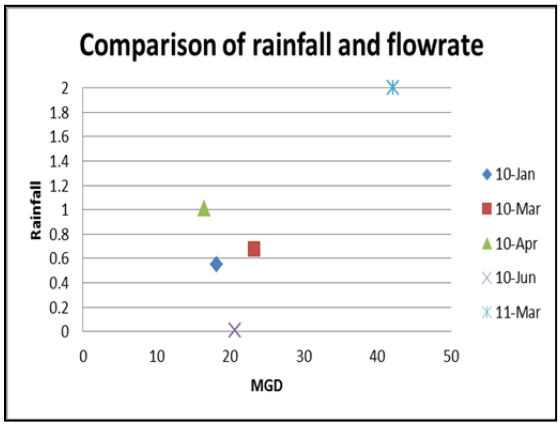


Figure E. Rainfall vs. Flow for Rain Events

There was a higher flow rate for the treatment plant during higher rainfall, particular for events with rainfall higher than one inch. From the infiltration of the stormwater, we want to determine if it has an effect on the treatability of the treatment. So, from the influent concentrations we calculated the mass that enters the treatment plant for each event.

Table 4. Example comparisons of influent PAH masses for dry and wet weather

Chemical	grams/day (wet weather) 03/02/10*	Mass/day (dry weather) 05/11/11**
sulfamethoxazole	4100	26
trimethoprim	1300	310
carbamazepine	1400	0.0
fluoxetine	360	0.0
acenaphthylene	150	3.0
acenaphthene	150	53
phenanthrene	110	140

*The recorded rainfall on 03/02/10 was 17 mm, and the flow rate was recorded as 23.3 MGD.
** the recorded flow rate on 05/11/11 was 13.5 MGD.

There is a higher mass content in the influent for all of the PAHs and pharmaceuticals during wet weather, except for phenanthrene. Carbamazepine and fluoxetine were not detected in the influent during the dry weather. PAHs are mostly being delivered by stormwater, so the increase of PAHs in the wet weather influent samples indicates that stormwater inflow affects the plant sewage, as expected from the increased plant treatment flows during wet weather, especially with rains greater than one inch (25 mm).

Wet weather concentrations for carbamazepine are shown in Figure F. These concentrations indicate an increase in during the treatment process. This is consistent with literature showing that carbamazepine is difficult to treat in wastewater treatment systems.

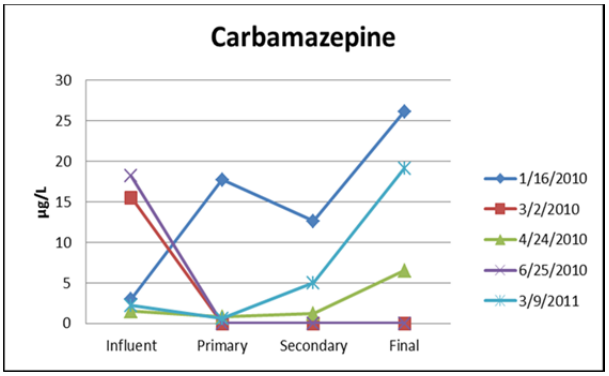


Figure F. Concentrations of carbamazepine

11.2.3 Conclusions

From our current data that has been analyzed, we found that there is an increase of the emerging pollutants entering the treatment plant, both PAHs and pharmaceuticals, during some of the large rain events. The PAHs showed higher masses in the influent during wet weather, but primary and secondary treatment was not affected. There were some effluents than were higher in concentrations than in the influent for some unit processes (especially for primary treatment), which may be due to wastewater matrix interference during the analyses. PAHs were consistent in showing significant reductions during both wet and dry weather which indicates that stormwater did not significantly affect treatability of these compounds.

For the pharmaceuticals, there was more variability in treatment for each constituent during wet weather. Ibuprofen and gemfibrozil showed a steady reduction in concentration, whereas carbamazepine showed no treatment. The difference in treatability may be caused by pharmaceuticals being active in wastewater by conjugation and deconjugation of metabolites, which may increase concentrations in the effluents. Wet weather samples will be com-

pared to dry weather samples to see if treatability is affected. Overall, it appears that pharmaceuticals were removed in the secondary treatment process compared to the other processes.

Final conclusions will be based on the complete data set, but these preliminary data indicate performance as expected, with minimal wet weather effects, although wet weather is shown to be a significant source of most of these pollutants.

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