

THESIS

GRAYWATER APPLICATION FOR LANDSCAPE IRRIGATION:
GREENHOUSE STUDIES

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ABSTRACT

GRAYWATER APPLICATION FOR LANDSCAPE IRRIGATION: GREENHOUSE STUDIES

Over the years, residential graywater application for landscape irrigation has garnered increasing popularity. Concerns however exist regarding the potential negative impacts that graywater pose to plant health and environmental quality. Due to the variability in field conditions such as graywater loading rate, soil type, climate and rainfall amount difficulty exists in accurately determining the potential for groundwater contamination. The need therefore arises to evaluate impacts of graywater irrigation in a controlled environment to develop scientifically justified conclusions regarding the fate of graywater constituents. The objective of this study was to conduct experiments in a greenhouse to evaluate the potential for groundwater contamination by conducting leachate analysis. Plant health was also evaluated throughout the duration of the experiment.

The experiment setup involved the use of thirty-eight custom polyvinyl chloride (PVC) pots. These pots were setup in the Colorado State University greenhouse. Potable water served as a control for the experiments. Two different plants and two different turfgrasses were utilized. The turfgrasses were bermudagrass (a warm season grass) and

tall fescue (a cool season grass). The landscape plants used were euonymous (a shrub) and lemon (a citrus). The pots were setup to allow for leachate collection from the bottom. The leachate volume was monitored and recorded and leachate analyses were conducted for boron, sodium adsorption ratio (SAR), nitrate, ammonium, total nitrogen (TN), total dissolved solids (TDS), total suspended solids (TSS), volatile suspended solids (VSS), total organic carbon (TOC), sulfate, conductivity and surfactants (linear alkylbenzene sulfonate (LAS), alkyl ethoxy sulfate (AES), and alcohol ethoxylate (AE)).

Analysis of the leachate from the graywater irrigated pots revealed on average, elevated levels of TOC, TN, nitrate, ammonium, TDS, TSS, VSS, sulfate, conductivity, boron and SAR when compared to the concentrations measured in the leachate from the control systems. The average concentrations of TOC, TSS, VSS, ammonium, nitrate and TN measured in the leachate from the graywater irrigated plant/grass systems were however lower than the concentrations in the synthetic graywater. An expected increase in conductivity and TDS in the leachate from the graywater irrigated pots was found. Results further indicate the accumulation of boron and salts (indicated by SAR) in the graywater systems with a trend of increasing concentrations with time and a subsequent increase in measured leachate concentrations above the input concentration measured in the graywater. With the exception of boron and salts, there was substantial percentage retention of graywater constituents through the soil column such that there was lower measured concentrations of the graywater constituents in the soil leachate compared to the input concentrations.

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Chapter 1

INTRODUCTION

1.1 Water Supply Limitations

Water is a valuable resource, and must be conserved. The need to ensure balance between water supply and water demand cannot be overemphasized. In both developing and developed countries, the dominance of water shortage is increasing. The need therefore arises to address the issue of adequate water supply to an ever-increasing population.

To combat the water shortage problem, possible remedies include water conservation and water reuse. Conservation encompasses a wide range of measures- from restrictions on water use (such as imposed regulations for residential landscape irrigation), the use of leak detection devices, to the use of water conservation devices such as faucet aerators, low flow showerheads and ultra low-flow toilets. Although water conservation serves as an efficient means of sustaining water resources, the focus in this study will be on water reuse.

Water reuse can be achieved through the use of graywater, reclaimed wastewater, or a combination of the two. Important aspects of water demand management include the recycling of wastewater and graywater, which leads to the preservation of high-quality fresh water, pollutant reduction in the environment and reduction in overall supply costs (Al-Jayyousi 2003).

Various parts of the world that are suffering from water shortage problems have a growing interest in water reuse. World regions suffering water scarcity such as the Middle East, Australia and southwest U.S. are the largest consumers of reused water (Exall et al. 2004). Water shortage problems in some cases can be attributed to high evaporation coupled with rainfall amounts that are low (e.g. Australia). Water shortage can also occur because of very high freshwater demands from the population (e.g. Japan) (Eriksson et al. 2002). In general, increase in water demand and changes in the climate are responsible for water supply problems (Surendran and Wheatley 1998).

In some cases, water reuse has resulted from worst-case scenarios in which the severity of water shortage prompted mandatory conservation actions. For instance, in Australia, severe water restrictions were imposed as a result of below average rainfall (Yiasoumi et al. 2008). These drought conditions in Australia have led to recognition by the Australian government on the local, state and federal level to establish policies and actions for conserving and recycling water (Pinto et al. 2010).

Water shortage problems serve as a motivation for water reuse. However, water reuse should not only follow the heel of a shortage problem, but should be pioneered based on the recognition of the value of water and the need to protect it as a resource for generations to come. This attitude is already being adopted in nations that are not facing an immediate need to reuse water. For instance, in Spain, there have been notable efforts towards water reuse (Van Riper and Geselbracht 1998). The view on water use can best be described with the words of Al-Jayyousi (2003) – “water is viewed as an economic good and a finite resource that should be valued and managed in a rational manner”.

1.2 Residential Water Use

On a residential scale, water that is of drinking quality standard is commonly used in the home for such purposes as lawn and garden irrigation, toilet flushing, car washing as well as other applications that do not require the use of such high quality water. The volume of water used per person in a household varies between developing and developed countries. In industrialized countries, in-house domestic water demand is approximately 100-150 liters per capita per day (l/c/d) (Friedler 2004). Okun (2000) states that water use in poorly developed cities is 100 liters (25 gallons) per capita per day but is as high as 600 liters (150 gallons) per capita per day in cities in more highly developed countries.

Within a country, the volume of water consumed will further vary based on several factors such as the geographic region and existing environmental conditions that can further affect water consumption in any given year. The residential water consumption in Fort Collins, Colorado in 2000, 2005 and 2009 was 211 gallons per capita per day (gpcd), 155 gpcd and 135 gpcd respectively (Larimer County 2009). The decline in water consumption between 2000 and 2004 was due to drought conditions and implementation of water use restrictions. Conversely, the water use by a family in Tuscon, Arizona is 102 gpcd with 61 gpcd used for indoor activities and 41 gpcd for outdoor use (Foster and DeCook 1986).

On a residential scale, water use can be divided into outdoor use and indoor use. In Denver, Colorado, in a given year, of the total water use, an average residential customer uses 55% for outdoor use such as landscaping (for the year), 11% for toilet flushing, 9% on showers and baths, 6% for faucets, 1% for dishwashing, 9% on laundry,

7% is lost as a result of leaks and 2% is used on other activities (Denver Water 2010). Similarly, in Cooper City, Florida, in a year, 54% of water is used for outdoor applications (Cooper City Utilities 2010). In dry climate, the outdoor water use can be even higher. The outdoor use is as high as 70% in Nevada (SNWA 2008). The American Water Works Association (AWWA 1999) estimates daily indoor water use as 69.3 gpcd (Table 1.1).

**Table 1.1 Indoor Residential Water Use
(AWWA 1999)**

Use	Gallons per Capita	Percentage of Total Daily Use
Showers	11.6	16.8%
Clothes Washers	15.0	21.7%
Dishwashers	1.0	1.4%
Toilets	18.5	26.7%
Baths	1.2	1.7%
Leaks	9.5	13.7%
Faucets	10.9	15.7%
Other Domestic Uses	1.6	2.2%

With over half of the potable water applied for irrigation and toilet flushing (Denver Water 2010), a substantial quantity of water can be conserved by using water of lower quality to meet these needs. Thus, consideration is given towards recycling water generated in a household to meet household water demand. The predominant water reuse options are use of reclaimed wastewater and graywater reuse.

1.3 Reclaimed Wastewater

Use of reclaimed wastewater for irrigation has become widely accepted. Wastewater reclamation involves treatment to a predetermined water quality that facilitates reuse, with the term-reclaimed water used to refer to treated effluent of a

quality suitable for a specific reuse application (Asano and Levine 1998). Reclaimed wastewater can be adapted for a vast range of uses. Uses of reclaimed wastewater include for lawn irrigation, industrial cooling towers, air conditioning, toilet flushing, dust control and construction (USEPA 2004). The supply of reclaimed wastewater for these uses is achieved through dual distribution systems (Okun 2000).

Although treated reclaimed water is a viable reuse option, on a residential scale, another option is to collect water that has been used (and in some cases treated prior to reuse) followed by direct application in the household to satisfy water demand within that household. Energy is required to pump and treat water as it travels from its source, to a home and then a sewage plant (Bennett 1995). Treatment of wastewater for distribution and use results in energy consumption and expense (Gelt 2007). However, graywater can be used with less treatment and thus results in energy savings (Gelt 2007). Therefore, to alleviate the increasing need and use of potable water, graywater application can be adopted.

Concerns exist regarding the reuse of graywater with regard to human health and the risk of soil and groundwater contamination as a result of the infiltration of graywater when it is reused for irrigation. Therefore, the objective of this study is to assess the potential for groundwater contamination as a result of graywater application for landscape irrigation. In particular, constituents in graywater (Section 1.4.5) were evaluated. These constituents include nutrients, boron, and organics such as surfactants. In order to adequately understand the concerns regarding graywater use, it is important to review how graywater is defined, the practicality of graywater use, its composition and previous findings and inferences on the fate of graywater constituents. Extensive review

on the presence and fate of pathogens is however not provided because pathogen presence and contamination potential of pathogens were not evaluated in this study.

1.4 Graywater

1.4.1 Definition

Graywater is defined as water that is collected separately from sewage flow that originates from clothes washers, bathtubs, showers and sinks, but does not include wastewater from kitchen sinks, dishwashers, or toilets (Al-Jayyousi 2003). In some cases, graywater is defined as including input from kitchens. Madungwe and Sakuringwa (2007) define graywater as consisting of all non-toilet household wastewater from showers, baths, hand basins, washing machines and kitchen sinks. However, the graywater definition that is adopted in this study does not include contributions from kitchen sources due to high organic content and presence of food borne pathogens in this water, which render the water unsuitable for reuse applications without extensive treatment.

1.4.2 Quality

The composition and thus quality of graywater is variable. Graywater source, and the type and duration of storage affect the composition of graywater with chemical compounds present originating from household chemicals, washing, piping and cooking (Eriksson et al. 2002). A storage tank used can lead to the presence of metals such as zinc and copper in the graywater (Christova-Boal et al. 1996). It is therefore important to have

an adequate assessment of the composition of graywater to evaluate potential human and environmental health risks.

Lack of information on the chemical and microbial quality of graywater limits adequate assessment of associated health risks from graywater use (Karpiscak et al. 1990). In addition to the previously mentioned factors, the graywater quality can also be affected by household activities that can further lead to the introduction of pathogens to graywater. Therefore, a consideration that is given in graywater use is the constituents present in the graywater and the significance of these constituents as related to human health and the environment. Given the significance of evaluating the graywater quality, a further review of the graywater constituents is provided (Section 1.4.5).

1.4.3 Uses

Different countries have varying reasons for the choice of graywater reuse with the reuse option driven by the demands of high population density but small land space in Japan unlike the US, Saudi Arabia, Jordan and Australia where reuse is in response to drought conditions (Al-Jayyousi 2003).

There are various uses of graywater both indoors and outdoors. Graywater can be reused for indoor applications such as toilet flushing and outdoor applications such as residential landscape irrigation. In Australia, there is a demand on water authorities to allow graywater reuse for the purposes of toilet flushing, as well as lawn and garden watering where there is a potential to reduce potable water usage (Jeppesen 1996).

In using graywater for toilet flushing, treatment first has to be done and standards have to be met. Coliform counts are measured in graywater; therefore, there is substantial

treatment requirement to meet the specified guidelines on thermotolerant coliform counts (Ottoson and Stenstrom 2003). Graywater systems therefore constitute a significant risk theoretically due to the presence of pathogenic agents (Albrechtsen 2002). Toilet bowls supplied with graywater have been found to have elevated levels of *E.coli* and *Enterococcus* (Albrechtsen 2002). Due to the higher human exposure risk associated with graywater use for toilet flushing, graywater use for only landscape irrigation is more simple and practical for an average residential user.

A large volume of water is used for residential irrigation, particularly in arid climates (Section 1.2). Therefore, substantial reduction in water demands can be achieved by reusing graywater for landscape irrigation. This further leads to reduction on the demands for potable water supplies (Pinto et al. 2010).

Aside from residential use, graywater can be adapted for commercial uses. Other uses of graywater include irrigation of golf courses, parks and other open spaces, and industrial evaporative cooling (Okun 1997). Graywater can also be used for groundwater recharge (Al-Jayyousi 2003). In areas where the groundwater table is high, graywater can recharge the water table when applied in excess with increased infiltration from graywater uses leading to improved aquifer recharge (Madungwe and Sakuringwa 2007). However, care has to be taken when applying graywater for this purpose. The Honolulu Board of Water Supply (HBWS) in utilizing recycled water over potable water aquifers adopted a cautious approach due to concerns of potential effects on groundwater (Muirhead et al. 2003).

Given the practicality of graywater use for residential landscape irrigation, and the potential risks to the environment from its use for this purpose, the focus of this study is

on graywater use for landscape irrigation.

1.4.4 Economic Benefit

Graywater makes up about 55% of the total wastewater in a home (AWWA 1999). Madungwe and Sakuringwa (2007) also state that graywater accounts for 60% of the total wastewater generated in homes. The reuse of graywater results in the reduction of potable water use.

Water conservation benefits include savings of 21% and 20% when graywater is only used for landscape irrigation and toilet flushing respectively with a combined saving of 31% of the total water use when graywater is used for both landscape irrigation and toilet flushing (Christova-Boal et al. 1996). The in-house net water consumption can be reduced by 40-60 l/c/d by using graywater for toilet flushing and in turn lead to a reduction in the urban water consumption by 10-20% (Friedler 2004). Similarly, Jefferson et al. (2001) found that 30% of the potable water could be saved by either reusing graywater for toilet flushing or recreational irrigation. Savings of up to 50% have been reported when domestic graywater is reused for irrigation of lawns and ornamental gardens (Jeppesen 1996). When graywater is used, there is subsequent reduction in the total volume of wastewater that is sent to the wastewater treatment plant. Therefore, an additional benefit of graywater use is the reduced load on wastewater treatment plants.

In addition to the preservation of natural resources, water reuse can also offer economic benefit. Water reuse can lower the total costs associated with wastewater handling, therefore, water reuse in some countries is driven by economics as well as environmental considerations (Eriksson et al. 2002). The need for increased capital

investment in municipal water distribution systems and energy costs may be reduced in addition to conserving municipal water for residential consumption as a result of the use of an effective graywater system in addition to other water-saving devices (Rose et al. 1991).

Graywater use is not limited to technologically advanced countries, but is also viable in the developing world. There is potential for graywater reuse in developing countries given recent developments in technology and changes in attitudes towards water reuse (Al-Jayyousi 2003). In domestic residences in the Middle East, the largest potential source of water savings can be achieved through graywater use (Al-Jayyousi, 2003). Saudi Arabia, Cyprus and Jordan have introduced graywater systems to optimize water use at the regional level (Al-Jayyousi 2003).

In certain regions, graywater use goes beyond the fulfillment of water conservation or economic benefit and is in reality vital to sustaining human life. For instance, in Harare, Zimbabwe, food security can be achieved through graywater irrigation (Madungwe and Sakuringwa 2007). In Palestine, graywater is utilized in the production of crops and citrus fruits (Madungwe and Sakuringwa 2007).

1.4.5 Composition

1.4.5.1 Constituents

The main constituents of concern in graywater are macronutrients, metals, organic chemicals and pathogens. Nutrients of concern include nitrates and phosphates. High concentrations of sodium are sometimes measured (Table 1.2) with contributions attributed to the use of detergents that contain sodium salts. Calcium, magnesium,

potassium and sulfates are also found in graywater. Some detergents contain boron and this can be harmful to plant health even at low concentrations (Madungwe and Sakuringwa 2007).

The presence of pathogens in graywater must also be considered. Fecal contamination can occur in graywater as a result of household activities that can include washing clothes contaminated with fecal matter in the laundry. The determination of pathogen presence is done through analysis for indicator organisms such as fecal coliform and fecal streptococci. Concerns exist that high enough fecal contamination levels can lead to adverse health effects. However, there is also controversy regarding the use of fecal coliforms as indicator organisms. It has been found that bacterial indicator densities overestimate fecal loads, which are attributed to the regrowth of coliform indicator organism within the graywater system; thus, the associated health risk is overestimated (Otto and Stenstrom 2003). Therefore, the fecal coliform count might not necessarily indicate the accurate presence of pathogens.

Given the variation that can occur in graywater composition (as discussed above and in Section 1.4.2); it is important to understand the graywater constituents. Therefore, a summary of common graywater constituents and measured concentrations is provided (Table 1.2) based on a literature review of graywater constituents carried out by Roesner et al. (2006).

Table 1.2 Graywater Constituents
(Adapted from Roesner et al. 2006)

Reference	Eriksson et al. (2003)	Rose et al. (1991)				Casanova et al. (2001a)
	Composite	Shower	Laundry Wash	Laundry Rinse	Composite	Composite
Temperature (°C)	21.6-28.2					
pH	7.6-8.6				6.54	7.47
COD (mg/L)	77-240					
BOD (mg/L)	26-130					64.85
TSS (mg/L)	7-207					35.09
Turbidity (NTU)		28-96	39-296	14-29	76.3	43
NH ₄ -N (mg/L)	0.02-0.42	0.11-0.37	0.1-3.47	0.06-0.33	0.74	
NO ₃ -N (mg/L)	<0.02-0.26				0.98	
Total-N (mg/L)	3.6-6.4				1.7	
PO ₄ -P (mg/L)					9.3	
Tot-P (mg/L)	0.28-0.779					
Sulfate (mg/L)					22.9	59.59
Chloride (mg/L)					9	20.54
Hardness (mg/L as CaCO ₃)					144	
Alkalinity (mg/L as CaCO ₃)					158	
Ca (mg/L)	99-100					
K (mg/L)	5.9-7.4					
Mg (mg/L)	20.8-23					
Na (mg/L)	44.7-98.5					
Total bacterial pop. (CFU/100mL)	4.0 x 10 ⁷ - 1.5 x 10 ⁸	1.0 x 10 ⁷ - 1.0 x 10 ⁸	1.0 x 10 ⁷ - 1.0 x 10 ⁸	1.0 x 10 ⁷ - 1.0 x 10 ⁸	6.1 x 10 ⁸	
Total coliform (CFU/100mL)	6.0 x 10 ³ - 3.2 x 10 ⁵	1.0 x 10 ⁵	199	56	2.8 x 10 ⁷	8.03 x 10 ⁷
Fecal coliform (CFU/100mL)		6.0 x 10 ³	126	25	1.82 x 10 ⁴ - 7.94 x 10 ⁶	5.63 x 10 ⁵
Fecal Streptococci (CFU/100mL)						2.38 x 10 ²
E. Coli (CFU/100mL)	<100-2800					

1.4.5.2 Surfactants

To adequately understand impacts on the soil matrix and plant health, it is important to review surfactants in detail and assess their given properties and associated effects. Surfactants (surface-active agents) account for the highest concentration of synthetic organic constituents in domestic wastewater (Shafran et al. 2005). Surfactants present in household graywater result predominantly from the use of soaps and detergents. Surfactants are organic molecules consisting of a hydrophilic head and a hydrophobic tail (Karsa and Porter, 1995). The hydrophobic group in surfactants contains alkyl chain lengths of C₁₀ to C₂₀ and the hydrophilic group is either polarized or has an electrical charge with the ability to form hydrogen bonds (Shafran et al. 2005). Surfactants are classified based on the hydrophilic group (head group) as anionic (negatively charged head group), cationic (positively charged head group), nonionic (no charge on the head group), or zwitterionic (head group with both positive and negative charges) (West and Harwell 1992). Of the synthetic sulfonate surfactants, alkylbenzene sulfonates are the most prominent (Lange 1999).

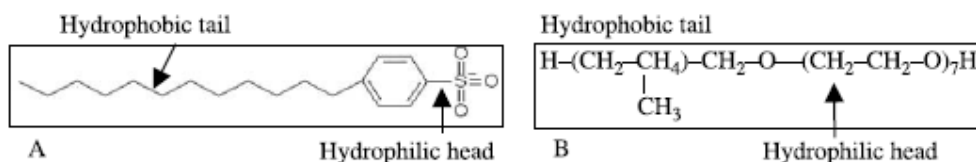


Figure 1.1 (A) Linear alkylbenzene sulfonate (anionic surfactant) and (B) Linear primary Alcohol Ethoxylate (nonionic surfactant) (Shafran et al. 2005)

The most commonly used commercial surfactants are linear alkylbenzene sulfonates (LAS), alkyl (or alcohol) ethoxy sulfates (AES), alkyl sulfates (AS), alkylphenol ethoxylates (APE), alcohol ethoxylates (AE), and quaternary ammonium compounds

((QAC)- a cationic surfactant) with LAS being the most popularly used anionic surfactant (Ying 2006). For this study, the surfactants of concern are LAS, AE, and AES given that these surfactants are common in household detergents and thus present in graywater. These surfactants are also the highest volume surfactants currently being used in commerce (McAvoy et al. 1998)

AE are a class of nonionic surfactants that are composed of a long-chain fatty alcohol with an ether linkage to a chain of ethylene oxide (EO) with 7 to 10 being the average number of EO units with a common range of 12 to 15 carbon units for the alkyl chain length for commercial grade materials (McAvoy et al. 1998). AES also have 12 to 15 carbon units but have EO groups range from 0 to 4 units and are anionic (McAvoy et al. 1998). LAS are also anionic surfactants but have 10 to 14 carbon units (McAvoy et al. 1998).

1.4.6 Exposure Consideration

Contamination potential spans from the presence of surfactants, boron, nitrogen as well as other macronutrients in graywater. Contamination concerns are particularly prevalent in cases where graywater is not treated before reuse. In dealing with the human risk aspect, measures can be taken to minimize or eliminate exposure risks. The safest route is to prevent human contact with graywater. This can be in the form of subsurface irrigation in which the graywater does not reach the ground surface, and surface irrigation in areas that are not habited (Jeppesen 1996). Subsurface irrigation such as drip irrigation systems can help minimize human exposure to graywater. Subsurface irrigation is not only beneficial to assuring human health but plant health as well. Given that 70% of a

plant's water and nutrition uptake takes place in the upper 50% of the root zone (for most plants in the top 30-60 m of soil), subsurface drip irrigation can be up to 60% more efficient than conventional surface spray by directly applying nutrient rich graywater to the root zone (Jeppesen 1996).

Though the human health risk can be minimized, consideration however still has to be given to the fate of graywater constituents in the soil given that soil contact with these constituents is unavoidable unlike human contact. Infiltration of graywater occurs after use for irrigation purposes. Therefore, long-term effects of graywater to the environment must be assessed.

The fate of metals and organics is important to consider. In relation to graywater constituents, metals detected in graywater are few. Of significance is the presence of organics such as surfactants, which can affect soil and plant health and have to be considered for their possible migration to groundwater.

1.4.7 Fate of Surfactants

It is important to understand the behavior of surfactants in soil. However, the fate of surfactants is not clearly understood. This can be attributed in part to the little information available regarding the impact of surfactants on the environment (Shafran et al. 2006). There is a lack of information available on surfactant biodegradation rates, which are important in evaluating human health risk with chronic exposure affected by the balance between loading rate and degradation (Garland et al. 2000). High adsorption of surfactants in soil will lead to lower concentrations in leachate while biodegradation

can also lead to lower measured surfactant concentrations in leachate. If the surfactant is strongly adsorbing, degradation is limited (Shafran et al. 2006).

The accumulation of surfactants is affected by the sorption and degradation characteristics. The adsorption capacity for surfactants varies and is affected by not only the surfactant properties but environmental conditions as well. The density of a surfactant will determine how it is affected by gravitational forces, which in turn can influence infiltration capability (Abu-Zreig et al. 2003). Uptake of surfactants at lower concentrations and the maximum plateau adsorption is affected by the degree of ethoxylation of the surfactants with higher adsorption observed at lower concentrations for surfactants with the same chain length but higher ethoxylation (Somasundaran et al. 1991). The adsorption of LAS on soil is dependent on the physical clay content with rapid LAS degradation in plant-soil systems, and its adsorption on soils involving both linear and exponential increasing isotherms (Ou et al. 1996).

1.4.7.1 Sorption of Surfactants

Anaerobic environments can affect biodegradability and subsequently sorption. Some surfactants are strongly adsorbing. Therefore, an area of concern is their adsorbing strongly in anaerobic environments (Steber et al. 1995). It is important to consider the distribution coefficient values (K_d). K_d is an indication of the sorption capability of a chemical to soil. It is an expression of the partitioning of an organic compound between the liquid and solid phase after the establishment of equilibrium (Doi et al. 2002). The distribution coefficient (K_{d1}) for LAS, AES and AE were determined using the KoWin program (McAvoy et al. 2002; Table 1.3).

Table 1.3 Surfactant Specific Parameters

Input Parameters		LAS	AE	AES
<i>Septic Tank</i>				
Surfactant usage (g/ca/d)		1.86	0.66	1.8
Anaerobic biodegradation rate constant, k_{b1} (day ⁻¹)		0.0	1.5 ^a	0.33 ^a
Distribution coefficient, K_{d1} (L/kg)		93 ^a	701 ^b	35 ^b
<i>Unsaturated Zone</i>				
Biodegradation rate constant, k_{b2} (day ⁻¹)	Aerobic	2.2	2.2 ^c	2.2 ^c
	Anaerobic	0.0	1.5 ^d	1.5 ^d
Distribution coefficient, K_{d2} (L/kg)	Range	1.2-4.0	9.1 ^b	0.5 ^b
	Mean	2.5		
<i>Saturated Zone</i>				
Biodegradation rate constant, k_{b2} (day ⁻¹)	Aerobic	0.47 ^e	0.5 ^c	0.5 ^c
	Anaerobic	0.0	1.5 ^d	1.5 ^d
Distribution coefficient, K_{d2} (L/kg)	Range	0.43-1.09	6.3 ^b	0.3 ^b
	Mean	0.84 ^f		

^a McAvoy et al. (2002)-simulation derived k_{b1} values

^b McAvoy et al. (2002)-estimated sorption coefficients (K_{d1} , K_{d2} , and K_{d3}) for LAS, AE and AES

^c Aerobic biodegradation rate constants identical to linear alkylbenzene sulfonate (LAS) in unsaturated and saturated soil zones [Knaebel et al. (1990), Itrich and Federle (1995), and Federle et al. (1997) (As cited in McAvoy et al. (2002)].

^d Anaerobic biodegradation rate constant for alcohol ethoxy sulfate (AES) as determined by Nuck and Federle (1996). An alcohol ethoxylate (AE) biodegradation rate was not available and therefore was assumed in McAvoy et al. (2002) to be similar to AES.

^e Mean value for site 2 in Doi et al. (2002)

^f Mean value for sites 2 and 3 in Doi et al. (2002)

K_{d1} is the distribution coefficient to suspended solids (L/kg), K_{d2} is the distribution coefficient for unsaturated soil (L/kg), and K_{d3} is the distribution coefficient for saturated

soil (L/kg). Soil from the study site was used to experimentally determine soil distribution coefficients (K_{d2} and K_{d3}) for LAS. Soil sorption coefficients (K_{d2} and K_{d3}) for AES and AE from published literature sources are provided in Table 1.3.

Doi et al. (2002) found that the K_d for LAS decreased with vertical distance below the ground surface with values decreasing from 4.02 L/kg to 0.43 L/kg. Ou et al. (1996) observed K_d values for LAS between 1.2 L/kg and 2.0 L/kg.

In a septic tank, sorption of surfactants to suspended solids will occur. There will likely be less biodegradation, thus prior to introduction to the environment, it can be expected that surfactants will have higher sorption coefficients. This correlates with values reported in Table 1.3. In the unsaturated zone, there is more time for biodegradation given that sorption results in longer residence time in this region unlike the septic tank where there is a short hydraulic retention time (McAvoy et al. 2002).

In addition, the soil type and organic carbon concentration will play a role in affecting the sorption of surfactants to soil (Table 1.4). An observation made in Table 1.4 is that with higher total organic carbon (TOC) concentration, K_d values are higher. This can be related to biodegradation (Section 1.4.7.2).

As shown in Table 1.4, for AE containing the same alkyl chain length, surfactants with higher EO numbers have higher sorption coefficients. Sorption also increased with increasing chain length. Given the dominance of the sorption behavior with alkyl chain length, it is likely that hydrophobic sorption mechanisms are occurring (Ying 2006). The anionic surfactant LAS also had lower K_d values than the nonionic surfactants. Sorption in the activated sludge is higher likely due to the presence of organic matter.

**Table 1.4 Sorption Coefficients of Surfactants
(Ying 2006)**

Compound	Sorbent	K _d (L/kg)	^a Reference
C12LAS	Sable soil (TOC 2.9%)	3.3-45	1
	Spinks soil (TOC 1.2%)	3.6-9	
	Sarpy soil (TOC 0.6%)	2-3.5	
C12LAS	Soil (TOC 0.46-1.08%)	1.2-2.0	2
^b C18TMAC	Activated sludge solids	1.8 x 10 ⁴	3
		4.9 x 10 ⁴	
^c NPEO3	River Sediment	1460	4
^c NPEO10	River Sediment	450	4
C15EO9	Sediment (TOC 0.2-2.8%)	350-2100	5
C1EO6	Sediment (TOC 0.76-3.04%)	40-62	6
C13EO3	Sediment (TOC 0.2-2.8%)	110-500	7
C13EO9	Sediment (TOC 0.2-2.8%)	110-590	7
C10EO3	Suspended Sediment (TOC 11%)	41	8
C10EO5		48	
C10EO8		126	
C12EO3		257	
C12EO5		724	
C12EO8		1230	
C14EO3		2951	
C14EO5		3467	
C14EO8		3548	

^a **References (as cited in Ying 2006):** (1) Orth et al. (1995), (2) Ou et al. (1996), (3) Games and King (1982), (4) John et al. (2000), (5) Cano and Dorn (1996a,b), (6) Brownawell et al. (1997), (7) Cano and Dorn (1996a,b), (8) Kiewiet et al.(1996).

^b**TMAC:** trimethyl ammonium chloride

^c**NP:** nonylphenol

1.4.7.2 Biodegradation of Surfactants

Biodegradation is used to describe the breakdown of an organic substance by living organisms. It is affected by environmental conditions. As previously mentioned, anaerobic environments can affect biodegradability. McAvoy et al. (2002) evaluated the fate of surfactants and found that AES and AS degradation are relatively unaffected by the degree of oxygenation of the soil unlike LAS which is not degradable under anoxic conditions, thus LAS will continue to be transported downgradient over time. Ying (2006) also acknowledges that LAS may be persistent in anaerobic environments. LAS has a half life of 7 to 33 days (Ying 2006), but a half life of no more than three days (first order biodegradation rate constant of $> 0.02/\text{d}$) would be required for LAS to be below detectable levels ($< 0.01 \text{ mg/L}$) to prevent entry into the groundwater for an unsaturated zone greater than 60 cm (McAvoy et al. 2002).

The long-term application of wastewater leads to increase in the concentration of less biodegradable surfactants in the soil-water environment (Abu-Zreig et al. 2003). There is a direct connection between the environmental fate of surfactants and their biodegradation behavior with the ecotoxicity being especially high in surfactants due to the resulting action against biological membranes because of the surface activity of these surfactants (Steber et al. 1995). The bioavailability of surfactants can be estimated from sorption data, with sorption having a notable effect on the degradation of the surfactant in the environment (Ying 2006). An important parameter for biodegradation is the biodegradation rate constant (k_b).

Estimates of the biodegradation rate (k_{b1}) (Table 1.3) were made for AES and AE (McAvoy et al. 2002). k_{b1} represents the first-order anaerobic biodegradation rate (d^{-1}),

k_{b2} is the first-order biodegradation constant in unsaturated soil (d^{-1}), and k_{b3} is the first-order biodegradation constant in saturated soil (d^{-1}). Given the lack of biodegradation of LAS in anaerobic environments as previously discussed, k_{b1} was assumed as $0 d^{-1}$. Soil from the study site was again used to experimentally determine the first-order soil biodegradation rate constants (k_{b2} and k_{b3}) for LAS. Soil biodegradation rate constants (k_{b2} and k_{b3}) for AES and AE from published literature sources are provided in Table 1.3.

Similar to the trend for sorption (Section 1.4.7.1), Doi et al. (2002) also found that the k_b for LAS decreased with vertical distance below the ground surface with values decreasing from $2.17 d^{-1}$ to $0.08 d^{-1}$.

Higher organic carbon removal is an indication of biodegradation. Therefore, when TOC is high (Table 1.4), there may be less organic carbon removal, thus less biodegradation (thus higher sorption leading to higher K_d values).

1.4.7.3 Implication of Surfactant Sorption and Biodegradation

Trends in the sorption and biodegradation parameters for anionic and nonionic surfactants are noted from Tables 1.3 and 1.4, and deductions can be made based on these trends. LAS has a higher sorption coefficient compared to AES, therefore, barring interference from plants, LAS will sorb more strongly to soil. Given that the biodegradation rates for these two surfactants are approximately equal, sorption will be the dominant mechanism that will determine the difference in concentrations of these surfactants that are measured in the soil system and leachate. AE has the highest sorption coefficient compared to LAS and AES and thus will have the highest sorption to soil. Therefore in a soil-water system, if equal amounts of LAS, AES, and AE are introduced,

the expected trend in surfactants in the soil would be AE>LAS>AES and thus in leachate it is expected that AES>LAS>AE. Soils with higher TOC would tend to indicate higher sorption and lower biodegradation. The soil type, ethoxylation of the surfactant and alkyl chain length will also affect sorption trends in a given soil system with sorption increasing with increasing alkyl chain length and EO group. For the analysis of leachate, the presence of suspended solids can also affect the measured surfactant concentrations given that sorption to suspended solids can occur.

1.4.7.4 Plant-Surfactant Interactions

The presence of plants can affect the fate of surfactants after graywater is applied for irrigation. Aquatic plants are known to take up xenobiotic compounds from solution and biotransform them in conjunction with their associated microbiota (Federle and Schwab 1989). Plant-surfactant interactions are important in not only considering possible phytotoxic effects of graywater on plants but also in considering the concentration of surfactants in soil leachate.

The internal dynamics of plants, plant physiology in addition to the effectiveness of microbial communities associated with the rhizosphere are important in graywater reuse (Pinto et al. 2010). The microbial activity in the rhizosphere that degrades surfactants and the use by plant for transpiration may be affected by the reuse of graywater for growing plants (Garland et al. 2000). Microbes present in the rhizosphere (the immediate narrow region around roots usually covered with a thin layer of soil) has a significant effect in degrading surfactants and other organic substances present in graywater (Pinto et al. 2010, Federle and Schwab 1989). LAS degradation has been found

to be faster in the rhizosphere when compared to surrounding sediments that were root free (Federle and Schwab 1989). The effectiveness of microbial communities associated with the roots of Walderman's Green lettuce, duckweed and roots of cattail in degrading alkylbenzene sulfonate and linear alkylbenzene sulfonate and eliminating the phytotoxic effects has been shown (Bubenheim et al. 1997, Federle and Schwab 1989).

Biological treatment of graywater under aerobic conditions occurs in the topmost region of the soil, which has the root zone of plants (Townshend 1993). If the system is not overloaded, soil and plants can process many of the contaminants present in graywater including sediment, nutrient, salt and organic material.

Conversely, it is possible that instead of degradation of surfactants occurring, the surfactants can exhibit toxic effects. Anionic surfactant content in graywater can alter the microbial communities associated with the rhizosphere and lead to phytotoxicity with the phytotoxic effects on different plants being highly variable (Pinto et al. 2010). Phytotoxic effects usually occur above 250 mg/L of surfactant (for instance for the surfactant igepon) (Bubenheim et al. 1997). The direct phytotoxic effect will be dependent on the rate of degradation of the surfactants as well as the toxic threshold of individual plants (Garland et al. 2000).

Potential soil impact results from surfactant accumulation in the soil that can alter the soil matrix (Shafran et al. 2005) as well as the accumulation of nutrients initially, followed by potential leaching beneath the soil profile. Although LAS has rapid degradation in plant-soil systems, its potential contamination of groundwater cannot be ignored given its strong penetration ability in soil coupled with its weak adsorption capability (Ou et al. 1996). Flow patterns and productivity of soils can be adversely

affected by surfactant accumulation (Shafran et al. 2006). Although accumulation of anionic surfactant in soil up to 30 mg/kg has been detected (Shafran et al. 2006), it is believed that surfactant concentrations in soil as a result of graywater irrigation have much lower concentrations.

The deduction regarding the concentration of surfactants in soil was made based on associated error with the testing method used by Shafran et al. (2006) and results from surfactant concentrations measured for graywater irrigated soils by the Sharvelle laboratory in residential locations. Surfactant concentrations measured in soils irrigated with graywater are lower compared to that measured by Shafran et al. (2006). The measured LAS concentration in graywater irrigated soil by Sharvelle et al. (2010) was 0.13 mg/kg. Of note also is that the testing method applied by Shafran et al. (2006)-MBAS (Methylene Blue Active Substances) is subject to interference and thus error in the measured concentrations. In measuring a particular MBAS such as LAS, positive interferences occur as a result of other MBAS, and negative interferences can also occur from the presence of particulate matter, cationic surfactants and other cationic materials (Ibrahim 2007). Further, the amount of methylene blue can be affected by other substances such as sulfates, organic sulfonates, carboxylates, chloride, nitrate, phenols, cyanates and inorganic thiocyanates (Ibrahim 2007).

Therefore, although surfactant accumulation in soil has been determined, the reported value in published literature has to be critically viewed and is believed to not accurately depict measured surfactant concentration as a result of graywater irrigation.

1.4.8 Fate of Other Graywater Constituents

Leaching of nutrients is also of concern and can be affected by a variety of conditions. Asano and Levine (1998) identify nutrients of concern in wastewater as nitrogen and phosphorus. Leaching of phosphorus to groundwater in clay soils may occur if the soil becomes phosphate-saturated with this problem being even more prevalent in sandy soils (Christova-Boal et al. 1996). In the case of surface application, there is the possibility of nitrogen leaching to the groundwater (Exall et al. 2004).

Removal mechanisms for contaminants include volatilization, precipitation of metals, sorption within the soil matrix and chemical or biological conversion reactions (Asano and Levine 1996). Despite the removal mechanisms, there is still the potential for constituents to reach groundwater aquifers. Most removal of chemical and biological constituents occurs in the top 2m of the vadose zone, however, nitrification and denitrification in addition to biological breakdown of organics occurs in the unsaturated zone and in the aquifer (Asano and Levine, 1996).

Based on the removal mechanisms in soil, inference has to be made regarding the accumulation and potential leaching of constituents like nitrogen and phosphorus. When plants are present, plant uptake of nutrients can lead to reduction of measured nutrients in the soil. There are varied results regarding the accumulation of nutrients in graywater irrigated soil in published literature. No significant increase in total nitrogen and total phosphorus has been found after plant harvesting following irrigation with graywater (Pinto et al. 2010). Pinto et al. (2010) found that the total nitrogen and total phosphorus levels in soil before initiation of graywater irrigation were respectively 324 mg/kg and 103.4 mg/kg. Following graywater irrigation, these nitrogen and phosphorus levels

measured by Pinto et al. (2010) were 347.8 mg/kg and 126.6 mg/kg respectively. In considering the same study by Pinto et al. (2010), measured total phosphorus concentrations after graywater irrigation on only soil with no plants was 153.8 mg/kg. This higher total phosphorus in the soil with no plants compared to the concentration measured in the soil when plants were present is indicative of the uptake of nutrients by plants that can occur. In contrast to the study by Pinto et al. (2010), accumulation of total nitrogen has been reported (Shafran et al. 2006). Accumulation of up to 385 mg/kg has been reported by Shafran et al. (2006) in soils irrigated with graywater that had a graywater total nitrogen concentration of 19 mg/L. The total nitrogen measured in soils irrigated with graywater is considered substantial given that freshwater irrigated soils only had 70 mg/kg total nitrogen (Shafran et al. 2006).

Boron accumulation has also been observed in graywater irrigated soils. Shafran et al. (2006) measured accumulation of boron of up to 0.3 mg/kg in soil, which is notable given soil concentrations of boron of 0.05 mg/kg prior to graywater irrigation. Gross et al. (2005) measured boron in graywater irrigated soil at levels up to 2.5 mg/kg.

A noteworthy consideration other than constituent accumulation is the effect of graywater on soil chemistry. Substantial effects on the soil pH and electrical conductivity (EC) have been observed following irrigation with graywater (Pinto et al. 2010), where changes in pH from 6.9 to 7.9 and in the EC from 126.2 $\mu\text{S}/\text{cm}$ to 306.3 $\mu\text{S}/\text{cm}$ as a result of graywater irrigation was found.

While effects of graywater to soil quality have been studied, the potential for graywater constituents to migrate to groundwater has not been evaluated. In the past 20 years, there has been an increased recognition of the need to ensure that the impact of

recycled water applications on soil and groundwater are environmentally sustainable in the long term (Anderson et al. 2001). Considered factors include nutrient loading rates, salt, chemical content, and hydraulic loading rates. In addition to these, long-term effects on plant health are important to understand.

1.4.9 Graywater Effect on Plant Health

Potential risks to plant health associated with graywater irrigation stem from elevated pH, salinity, and boron with the possibility of accumulation of pathogens, metals and organic chemicals in receiving soils (Finley et al. 2009). Therefore, to prevent possible plant damage, guidance for graywater reuse for irrigation is required given the possible presence of boron and surfactants (Townshend 1993). Even in very low concentrations, boron can cause plant damage, and sulfates present in many surfactants can be injurious to plants at high concentrations (Townshend 2003).

The potential exists for graywater to increase the soil alkalinity if applied for irrigation over a long period. The reuse of graywater with a pH in excess of 8 can lead to increased soil pH and subsequent reduction in the availability of some plant micronutrients with a resulting effect on plant growth (Christova-Boal et al. 1996).

In making assessments on plant effects, several factors such as observed die back from the leaf tip, chlorosis and changes in biomass have to be considered. Similar to the inconsistencies in reports of nutrient accumulation, there are also varied reports on the impact of graywater to plant health. Pinto et al. (2010) found no significant effects on plant dry biomass for silverbeet, water use or number of leaves as a result of graywater irrigation. On the other hand, Shafran et al. (2006) found adverse effects on plant health

given the development of chlorosis after graywater irrigation that occurred in the plants studied as a result of elevated salinity and boron levels. Toxicity to algae and willow trees from kitchen and laundry graywater and toxicity of bathroom graywater to only algae have been found (Eriksson et al. 2006).

Ayers and Westcot (1985) state that common toxic ions present in irrigation water are sodium, boron and chloride with damage occurring either as a result of the individual ions or a combination of several ions. Chloride is not adsorbed by soils, and therefore commonly results in toxicity with sensitivity to toxicity varying based on the plant type (Ayers and Westcot 1985). For citrus, the variation of acceptable chloride levels in the irrigation water is from 6.7 me/L (237 mg/L) to 16.6 me/L (588 mg/L) with more sensitive crops like avocados having a threshold of 3.3 me/L (117 mg/L) to 5.0 me/L (177 mg/L) and less sensitive crops like grapes having a threshold as high as 27 me/L (956 mg/L) (Ayers and Westcot 1985). The concentrations for chloride provided by Ayres and Westcot (1985) above are given as milliequivalents per Liter (me/L). The corresponding concentrations in mg/L were computed and provided in parenthesis beside the values in me/L provided by Ayers and Westcot (1985).

Boron is essential for plant growth and is needed in small amounts but becomes toxic at amounts greater than what is required by the plant. Even in very low concentrations, boron can cause plant damage with tolerance in the range of: 0.5 to 1.0 mg/L for sensitive crops (slight to moderate damage), 1.0 to 2.0 mg/L and 2.0 to 4.0 mg/L for semi-tolerant and tolerant crops respectively (Townshend 1993). Townshend (1993) further asserts that sulfates can be injurious to plants at high concentrations and

recommends that a limit of 500 mg/L be set for lawn irrigation with a chloride limit also recommended as 500 mg/L.

A summary of acceptable levels of SAR, EC and nutrients are provided (Table 1.5). These values are representative of acceptable levels for adequate water quality in terms of influence on plants and soil conditions.

Table 1.5 Guidelines for Water parameters and Ion Concentrations
(Ayers and Westcot 1985)

Criteria Evaluated	Degree of Restriction on Use		
	None	Slight to Moderate	Severe
<i>Salinity</i>			
EC (dS/m) OR	< 0.7	0.7 - 3.0	> 3.0
TDS (mg/L)	< 450	450 – 2000	> 2000
SAR = 0 – 3 and EC =	> 0.7	0.7 - 0.2	< 0.2
SAR = 3 – 6 and EC =	> 1.2	1.2 - 0.3	< 0.3
SAR = 6 – 12 and EC =	> 1.9	1.9 - 0.5	< 0.5
SAR = 12 – 20 and EC =	> 2.9	2.9 - 1.3	< 1.3
SAR = 20 – 40 and EC =	> 5.0	5.0 - 2.9	< 2.9
<i>Ion Toxicity</i>			
Sodium (surface Irrigation) (SAR)	< 3	3.0 - 9.0	> 9
Sodium (sprinkler Irrigation) (me/L)	< 3	> 3	
Chloride (surface Irrigation) (me/L)	< 4	4.0 -10.0	> 10
Chloride (sprinkler Irrigation) (me/L)	< 3	> 3	
Boron (mg/L)	< 0.7	0.7 - 3.0	> 3.0
<i>Others</i>			
Nitrogen (NO ₃ ⁻ -N) (mg/L)	< 5	5.0 -30.0	> 30
Bicarbonate(HCO ₃ ⁻) (me/L)	< 1.5	1.5 - 8.5	> 8.5
pH	Normal Range 6.5-8.4		

1.5 Summary

Given the knowledge that graywater quality and quantity is affected by numerous factors including the graywater source and personal care products used in a home, it is important to evaluate how the use of graywater for residential landscape irrigation will affect the soil and plant life as well as overall long-term environmental impacts. Long-term irrigation of arid soil with graywater can cause accumulation of salts, surfactants and boron in the soil thus leading to plant toxicity and changes to soil properties (Gross et al. 2005).

The most prominent mechanism for the complete removal of chemical substances from the soil and aquatic environment is biodegradation with the environmental impact further governed by ecotoxicity (Steber et al. 1995). The properties of the soil environment and chemical degradation rates will affect the transport of graywater constituents. Studies have not been done to adequately assess the fate of graywater constituents in the soil depth at representative distances to the groundwater table. Researchers have reported vastly different results regarding accumulation of nutrients, and the impacts of graywater irrigation to groundwater are very unclear.

Guidance must be provided for safe graywater irrigation. There is an immediate need to adequately assess and quantify the environmental effects of graywater. At this time, knowledge gaps still exist regarding long-term effects of graywater. These knowledge gaps result from inconclusive findings regarding the fate of nutrients and surfactants in soil. In addition, no conclusive data is available regarding the potential for graywater constituents to migrate to groundwater.

Thus, in general, research has to be done to determine what long-term graywater effects to the environment and subsequently to human health can occur. Precise scientific justifications are difficult to make in the field due to the variability in field conditions such as the graywater loading rate and composition. The presence of pets can cause inconsistency in findings regarding fecal contamination and rainfall can cause dilution effects on measured constituents that have undergone infiltration. The relevancy of this study is not only important from a human health and environment perspective, but serves a crucial role to aid in graywater legislation. With greater understanding of graywater effects, more states can harness graywater as an effective water conservation means.

1.6 Objective of the Study

The objective of this study is to evaluate the potential for graywater contaminants to migrate to groundwater by conducting analysis of leachate produced in plant pots through greenhouse studies. Graywater and potable water were applied to different plant and grass types. The leachate was analyzed for boron, sodium adsorption ratio, sulfate (as sulfur), nitrate, ammonium, total nitrogen, total dissolved solids, total suspended solids, volatile suspended solids, total organic carbon, conductivity, and surfactants (LAS, AES, and AE). A visual evaluation of plant health was made. However, detailed testing and evaluation was not carried out. Therefore, precise scientific findings regarding effect of graywater on plant health were not made in this study. To provide known contaminant loading rates and ensure consistency and repeatability of the results, synthetic graywater was applied to the plants.

A hypothesis was developed (provided below) regarding the fate of graywater constituents in this study based on the reviewed literature on measured graywater constituent concentrations, and the behavior of such constituents such as the sorption and biodegradation behavior of surfactants.

Given the leaching behavior of nitrogen, it is expected that concentrations of this nutrient measured in the leachate from graywater irrigated soils will be elevated compared to nitrogen measurements in control scenarios where leachate from potable water irrigated soils are measured. However, given the removal mechanisms that occur as a result of plant uptake, the nitrogen measured in the leachate from graywater irrigated soils will be less than the measured nitrogen concentration in the graywater. For cases where plants are not present, and there is therefore the elimination of nutrient uptake by plants, higher levels of nitrogen will be measured in the leachate from soils with no plants compared to cases where plants are present.

Boron accumulation has been determined in graywater irrigated soil. It is therefore expected that boron will also accumulate in the graywater irrigated soils in this study. Therefore, the boron measured in the soil leachate will indicate accumulation of boron in the soil with time.

With high contributions of sodium from graywater, along with salt contributions, as is typically the case in residences, it is expected that elevated salt levels and dissolved solids will be detected in measured leachate concentrations. Elevated EC is also expected given the effects that graywater can have on soil alkalinity and the increased salt content in the soil that will occur as a result of graywater irrigation.

Based on the sorption and biodegradation behavior of LAS, AE and AES, it can be stipulated that these surfactants will have lower concentrations in the soil leachate compared to the concentrations in the graywater. LAS use is typically higher compared to AES and AE (Section 1.4.5.2) and this leads to higher LAS concentrations often measured in the field (Chapter 2 – Section 2.3.3.1). Given that simulated conditions in this study will consist of higher LAS concentrations relative to AES and AE, coupled with the use of sandy loam soil in this study, it is believed that higher LAS will be measured in the leachate compared to AES and AE.

These above hypotheses will form the basis for further evaluation in this study and will be important for proceeding with developing the hypothesis for statistical analysis.

The thesis was organized in order to first identify the need to study the effects of graywater application and thus validate the need for the greenhouse studies (Chapter 1). Following this review, a good foundation for the introduction to the research project was established followed by a description of the experiment setup, analysis methods, results and finally inferences that can be made based on the research findings.

Chapter 2 gives a more detailed description of various graywater definitions and graywater characteristics. In particular, the physical, chemical and microbial characteristics of graywater are considered, surfactant concentrations are reviewed and comparisons are made based on graywater composition as affected by the source. In Chapter 3, the experiment setup, materials and testing methods are provided. Finally, the research results, discussions of the results and a summary of the research findings are provided in Chapter 4.

Chapter 2

GRAYWATER DEFINITION AND CHARACTERISTICS

2.1 Objective

Concerns exist regarding the reuse of graywater with regard to human health and the risk of soil and groundwater contamination as a result of the infiltration of graywater after it is reused for irrigation. The graywater quality is variable based on the source, storage prior to use, in addition to other factors (Chapter 1 - Sections 1.4.2 and 1.4.5.1). Difficulty exists in accurately assessing potential groundwater contamination from graywater application in the field due to the variability in field conditions that exists such as the soil type and the effects of pets at the graywater application sites. The need therefore arises to make a scientifically justified assessment of the contamination potential from graywater application. In order to achieve this goal, it is important to not only eliminate the environmental impacts that affect accurate contamination potential assessment, but to also control the quality of the applied graywater.

Therefore, the objective of this study is to assess the potential for groundwater contamination as a result of graywater application for landscape irrigation by conducting analysis of leachate produced in plant pots through greenhouse studies. To provide known contaminant loading rates and ensure consistency and repeatability of the results, synthetic graywater was applied to the plants.

Other researchers have previously adopted the use of synthetic graywater. To achieve pH, electrical conductivity (EC), total nitrogen and total phosphorus values similar to averaged literature values, Pinto et al. (2010) used 0.7g of Spree Matic Concentrate (a commonly available detergent in Australia) in 1000 mL of potable water. Known quantities of soap, detergent, starch, yeast extract, cooking oil and settled sewage were used by Surendran and Wheatley (1998) in order to simulate graywater. Travis et al. (2010) used vegetable oil, laundry powder, pulverized bar soap, raw dining/kitchen effluent and potable water to create synthetic graywater.

In contrast to the above literature studies, the synthetic graywater in this study was not formulated using household products. To achieve exact constituent concentrations based on a predetermined target composition and eliminate added compound concentrations not accurately accounted for in synthesized products, laboratory chemicals, surfactants and yeast extract were mixed together using deionized water to formulate synthetic graywater (Chapter 3 - Section 3.5). It was therefore important to conduct a review of graywater constituent concentrations in addition to graywater physical and chemical characteristics as provided in literature in order to accurately determine the appropriate graywater constituents to simulate at suitable concentrations for the synthetic graywater recipe.

In this chapter, the various characteristics of graywater based on source are examined. From this review, the synthetic graywater could therefore be accurately formulated to represent concentrations of graywater constituents as would typically be expected in actual graywater.

2.2 Graywater Definition

Graywater (or greywater), sometimes referred to as “sullage” (Jeppesen 1996) or human hygiene water (Garland et al. 2000) is defined as wastewater without inputs from toilets, that is, wastewater from showers, laundry, bathtubs, hand basins and kitchen sinks (Ottoson and Stenstrom 2003). This definition has been widely adopted in literature including in Eriksson et al. (2002) where graywater is defined as wastewater originating from bathtubs, showers, hand basins, laundry machines and kitchen sinks without any input from toilets. Jeppesen (1996) refers to graywater as all other wastewater excluding blackwater (water from toilets and having high fecal contamination). Finley et al. (2009) also defines graywater as wastewater generated excluding toilet wastes.

Kitchen graywater is not recommended for inclusion in graywater reuse because it contains undesirable compounds, is highly polluting and accounts for only 5% of the average household consumption (Christova-Boal et al. 1996). Kitchen sinks are major pollutant contributors and contribute 58%, 42%, 48%, 43% and 40% of the total volatile suspended solids (VSS_t), total chemical oxygen demand (COD_t), total biochemical oxygen demand (BOD_t), total oil and anionic detergents respectively (Friedler 2004). In addition to kitchen sinks, dishwashers are also pollutant contributors and contribute substantial amounts of pollutants including phosphates and boron (Friedler 2004).

Therefore, to minimize health risks associated with graywater, wastewater from kitchen sources (sinks and dishwashers) are often excluded from the standard graywater definition. In appendix G of the 2007 California Plumbing Code, graywater includes water that has been used from bathtubs, showers, wash basins, bathroom sinks, clothes washing machines and laundry tubs, but does not include wastewater from kitchen sinks

or dishwashers. Surendran and Wheatley (1998) also describe graywater as water from washbasins, showers, baths and washing machines.

This research focuses on reuse of graywater for landscape irrigation with minimal treatment of the graywater. Therefore, given the risks associated with the inclusion of graywater from kitchen sources and the negligible volume generated from these sources, the graywater definition adopted in this study is “water that originates from bathtubs, laundry, and bathroom sinks but does not include water from kitchen sinks or dishwashers”.

2.3 Graywater Characteristics

2.3.1 General Overview

The characteristics of graywater are determined by several factors. The main constituents of concern are macronutrients, metals, organic chemicals and pathogens. High levels of aluminum, sodium, and phosphorus have been measured in graywater (Christova-Boal et al. 1996). Increased concentrations of ammonia and phosphates have also been found in graywater (Rose et al. 1991). Given that graywater is low in suspended solids and turbidity, it can be deduced that a higher percentage of graywater contaminants are dissolved (Al-Jayyousi 2003).

The quality of graywater will vary according to geographical location, the level of occupancy in a home, demographics in a home, and the graywater source (Al-Jayyousi 2003). Statistically significant differences for chlorides, sulfates, turbidity, pH, biochemical oxygen demand (BOD) and fecal coliforms were found between one household with two adults and another with two adults and one child (Casanova et al.

2001a). For a selected graywater source, variations in graywater composition can still occur. For example, based on the stages in the cycles of a washing machine, the level of contamination can widely vary (Friedler 2004). The turbidity of graywater obtained from laundry alone also varies based on whether the source is from the wash cycle or from the rinse cycle with the wash cycle having higher turbidity values (Eriksson et al. 2002).

Although the source of graywater within a home affects its quality, the concentration of metals and other elements in the graywater is also determined by the concentrations and quality of the water from the water works (Eriksson et al. 2002). Changes in water consumption and the subsequent amount of discharged substances lead to variation in graywater composition. Graywater quality can also change with time with substantial chemical changes occurring within just a few hours (Al-Jayyousi 2003). The methods of conveyance and storage of graywater could affect its quality. A one order of magnitude increase in total bacterial standard plate count and coliform bacteria was found to occur with storage (Rose et al. 1991). As a result of galvanized steel collection tanks and household plumbing, high levels of zinc and copper have been measured in graywater (Christova-Boal et al. 1996).

Chemical products used around the home for cleaning and personal care products can lead to the introduction of xenobiotic organic compounds (XOCs) to graywater (Eriksson et al. 2002). Therefore, an additional factor that has to be taken into account is the possibility of the accumulation of XOCs and metals in the environment (soil and groundwater; Eriksson et al. 2002).

The presence of salts is also of concern. Salt accumulation in soil can have detrimental effects. Salt accumulation can be evaluated based on the electrical

conductivity and sodium adsorption ratio. Salinity is expressed by the electrical conductivity (EC) and SAR is a measure of the exchangeable sodium content in soil (Faruqui and Al-Jayyousi 2002). The sodium adsorption ratio (SAR) is determined based on the concentration of sodium, calcium and magnesium in the graywater. High SAR values can have adverse effects on plant systems, physical properties of clay soils and their hydraulic conductivity (Christova-Boal et al. 1996). SAR values in raw graywater on average of 3.3 was measured by Al-Hamaiedeh and Bino (2010) with a range of 2.23 to 4.76. To maintain a healthy soil and plant environment, the EC and SAR have to be considered together with different thresholds on acceptable EC being affected by the measured SAR (Chapter 1 - Section 1.4.9). For example, in arid environments where soils are high in salinity, values of EC of less than 2 mS/cm are not considered harmful (Shafran et al. 2006).

The choice of cleaning products in homes is important in the reduction of the environmental impact of graywater (Madungwe and Sakuringwa 2007). Laundry graywater can lead to substantially higher SAR values, even in excess of 10 in graywater (Christova-Boal et al. 1996). Saline graywater is often produced as a result of washing powders in which sodium salts are utilized as bulking agents (Madungwe and Sakuringwa 2007). Elevated levels of sodium in laundry wastewater have been found compared to other graywater sources (Eriksson et al. 2002). Thus, higher SAR values have been found to correspond to higher detergent concentrations (Faruqui and Al-Jayyousi 2002). The negative impact that high SAR in graywater applied for irrigation can have cannot be ignored. When SAR values measured in graywater irrigated soil are high, the implication is that the sodium content in the soil is high as well. High sodium

concentration can lead to soil dispersion, therefore the SAR in graywater should be below 4.0 (Gross et al. 2005).

Given the wide variations in graywater composition that exists from the above mentioned influences, it is important to consider the characteristics of graywater. The importance of considering the characteristics of graywater lies in determining the potential for graywater reuse. It is therefore important to not only consider the chemical characteristics of graywater, but physical characteristics and constituent concentrations as well. Some graywater characteristic parameters evaluated include physical properties, chemical characteristics, microbial content, the concentration of nutrients and other constituent concentrations. Physical parameters include total dissolved solids (TDS), total suspended solids (TSS), volatile suspended solids (VSS), temperature, and turbidity. Chemical properties such as electrical conductivity (EC), total organic carbon (TOC), biochemical oxygen demand (BOD) and sulfate concentrations are evaluated. Review of nutrient concentrations of total nitrogen (TN), nitrate nitrogen ($\text{NO}_3\text{-N}$), ammonium nitrogen ($\text{NH}_4\text{-N}$), total phosphorus (TP) and phosphate-phosphorus ($\text{PO}_4\text{-P}$) are also included in addition to concentrations of boron and salts. Given that microorganisms are also present in graywater, the microbial content of graywater has also been reviewed.

2.3.2 Microbial Content

One of the obstacles to graywater reuse is pathogen content associated with the graywater. The presence of enteric organisms such as *Escherichia coli* in graywater serves as an indication of fecal contamination and possible intestinal pathogens such as enteric viruses or *Salmonella* (Rose et al. 1991). Coliform levels (total and fecal) can also

indicate pathogen contamination levels. Elevated coliform levels in graywater can lead to elevated levels in graywater irrigated soil as well. Casanova et al. (2001b) found statistically significant differences between graywater irrigated soil and soil not irrigated with graywater (background soil; Table 2.1).

Table 2.1 Geometric Averages of Fecal Coliforms in Soil
(Casanova et al. 2001b)

Factor	Fecal Coliforms (CFU/g dry soil)	
	Graywater Irrigated Soil	Background Soil
With Children	1.26×10^3	8.99×10^0
Without Children	3.24×10^1	4.07×10^0
Including Kitchen Sink	1.56×10^3	2.61×10^0
Excluding Kitchen Sink	2.69×10^1	8.25×10^0
In-Ground Storage	7.85×10^0	4.57×10^2
Above-Ground Storage	¹ NA	¹ NA
With Animals	1.72×10^2	5.88×10^0
Without Animals	1.88×10^2	1.05×10^1

¹NA-Not Applicable

Microbial content can vary based on several factors such as graywater source and household demographics. The total bacterial populations in graywater from shower water, laundry wash water and laundry rinse water on average have been found to be 10^7 - 10^8 colony forming units (CFU) per 100 mL (Rose et al. 1991). For families with no children, total coliforms and fecal coliforms were low (6 and 80 CFU/100 mL on average) while for families with young children substantially higher values were measured and averaged 1.5×10^3 and 3.2×10^5 CFU/100mL for fecal coliform and total coliform respectively (Rose et al. 1991).

2.3.3 Physical and Chemical Characteristics

Graywater has all the constituents of raw sewage and is thus considered as dilute sewage (Christova-Boal et al. 1996). Surendran and Wheatley (1998) found graywater characteristics similar to those of settled domestic wastewater except for the concentration of ammonia and the bacteria content.

Physical parameters that are important to consider in graywater include turbidity, color, suspended solids and temperature with higher temperatures usually being unfavorable given that microbial growth is favored at higher temperatures (Eriksson et al. 2002). The temperature of graywater is affected by personal habits in a home. The use of warm water for personal hygiene can lead to graywater with high temperatures (in the range of 18°C to 38°C; Eriksson et al. 2002).

Some important chemical parameters to consider are the alkalinity, pH and hardness. The alkalinity, hardness and pH of the infiltrating graywater will affect the soil pH and buffering capacity (Eriksson et al. 2002). Important information can also be gleaned from the measurement of nutrients as well as the BOD and the chemical oxygen demand (COD). The risk of sulfide production can be assessed based on the possible oxygen depletion that can occur as a result of the degradation of organic matter during storage and transport (Eriksson et al. 2002). In irrigation waters, there are often regulations regarding the BOD and COD.

As previously mentioned, the graywater composition will vary based on its source and the hygiene products used. Surendran and Wheatley (1998) characterized graywater from university residence halls (Table 2.2) and found wide variations in composition

based on the amount of detergents and soaps used. The BOD and COD reported by Surendran and Wheatley (1998) show variations based on the graywater source.

**Table 2.2 Average Pollutant Concentration in Graywater
(Surendran and Wheatley 1998)**

Parameter	Source			
	Bath/Shower	Washbasin	Washing machine	Kitchen sink
pH	7.6	8.1	8.1	-----
NH ₄ -N (mg/L)	1.56	0.53	10.7	4.6
NO ₃ ⁻ -N (mg/L)	0.9	0.34	1.6	0.45
PO ₄ ³⁻ -P (mg/L)	1.63	45.5	101	15.6
Inorganic Carbon (mg/L)	26	20	25	-----
Total Organic Carbon (mg/L)	104	40	110	-----
Suspended Solids (mg/L)	76	40	68	-----
Dissolved Solids (mg/L)	559	520	590	-----
Volatile Solids (mg/L)	318	240	330	-----
BOD (mg/L)	216	252	472	536
COD (mg/L)	424	433	725	936
Turbidity (NTU)	92	102	108	-----
Total coliforms (CFU/100 mL)	6 x 10 ⁶	5 x 10 ⁴	7 x 10 ⁵	-----
Fecal coliforms (CFU/100 mL)	600	32	728	-----

It was found (Table 2.2) that contributions from the washing machines resulted in the highest measured concentrations for all the listed parameters in Table 2.2 when compared to the concentrations measured in the graywater from the washbasin and bath/shower sources. The exception to the trend of the higher measured concentrations in the washing machine graywater compared to the other sources occurred for the inorganic carbon, total

coliforms and suspended solids which were higher in the bath/shower graywater compared to the concentrations measured in the graywater from the washing machines.

Christova-Boal et al. (1996) also investigated various graywater source combinations to determine effects of different graywater sources on the characteristics of the graywater (Table 2.3). Similar to findings by Surendran and Wheatley (1998), the pH, alkalinity and presence of ions were affected by the products used in the bathroom or laundry. It was further found by Christova-Boal et al. (1996) that laundry water was the highest contributor of salts with high levels of sodium measured in graywater samples.

Table 2.3 Graywater Physical, Chemical and Microbial Characteristics
(Christova-Boal et al. 1996)

Parameter	Source	
	Bathroom water range	Laundry water range
pH	6.4-8.1	9.3-10
EC ($\mu\text{S}/\text{cm}$)	82-250	190-1400
Total Phosphorus as P (mg/L)	0.11-1.8	0.062-42
NH ₄ -N (mg/L)	<0.1-15	<0.1-1.9
NO ₃ ⁻ N (mg/L)	<0.05-0.20	0.10-0.31
Total Alkalinity as CaCO ₃ (mg/L)	24-43	83-200
Calcium (mg/L)	3.5-7.9	3.9-12
Magnesium (mg/L)	1.4-2.3	1.1-2.9
Sodium (mg/L)	7.4-18	49-480
Potassium (mg/L)	1.5-5.2	1.1-17
Suspended Solids (mg/L)	48-120	88-250
BOD (mg/L)	76-200	48-290
Sulfur (mg/L)	1.2-3.3	9.5-40
Chloride as Cl (mg/L)	9.0-18	9.0-88
Turbidity (NTU)	60-240	50-210
Fecal coliforms/100 mL ¹ MPN	170-3.3 x 10 ³	110- 1.09 x 10 ³
Total coliforms/100 mL ¹ MPN	500-2.4 x 10 ⁷	2.3 x 10 ³ - 3.3 x 10 ⁵

¹MPN = most probable number

Eriksson et al. (2002) noted that the COD of graywater from bathroom sources (184-633 mg/L) was less than the COD from laundry sources (725-1815 mg/L). In comparing BOD values, bathroom sources had 76-300 mg/L BOD while laundry sources had 48-472 mg/L (Eriksson et al. 2002). Similar to the BOD and COD, variation in the total organic carbon (TOC) also occurs based on the graywater source. Friedler (2004) found TOC in the range of 91 mg/L (in graywater from bathtubs) to 361 mg/L (in graywater from laundry sources). Faruqi and Al-Jayyousi (2002) determined the organic content in graywater (measured as BOD) in the range from 275 mg/L to 2,287 mg/L.

A comprehensive summary showing the variation of graywater physical and chemical characteristics, nutrients, salts, other constituent concentrations and microbial content from various literature sources is provided in Tables 2.4 and 2.5.

Table 2.4 Graywater Characteristics from Various sources (Part 1 of 2)

Parameter	Source			
	Finley et al. (1998) Shower, Bath and Laundry	Jefferson et al. (2001) Bath, Shower, Hand Basin	Ramon et al. (2004) Sports Center Public Shower	Pidou et al. (2008) Bath, Shower, Hand Basin from Residence Hall
pH	6.7-7.6	-----	7.5	6.6
NH ₄ -N (mg/L)	1.2-6.2	-----	2.7	0.7
Nitrate (mg/L)	-----	-----	0.67	3.9
Total Nitrogen (mg/L)	-----	5.0	-----	7.6
Sulfur (mg/L)	5.0-8.8	16.3	-----	-----
Sulfate (mg/L)	-----	-----	58.0	-----
Boron (mg/L)	-----	-----	0.13	-----
Phosphorus (mg/L)	0.24-1.02	1.37	-----	-----
Phosphate (mg/L)	-----	-----	0.09	0.5
Calcium (mg/L)	30-44	47.9	79.6	-----
Total Solids (mg/L)	313-543	-----	-----	-----
Magnesium (mg/L)	8.0-9.9	5.29	47.6	-----
Potassium (mg/L)	2.2-2.5	5.79	10.4	-----
Sodium (mg/L)	20-27	-----	106.0	-----
COD (mg/L)	278-435	-----	170	144
BOD (mg/L)	-----	-----	78	39
Total Suspended Solids (mg/L)	-----	-----	29.8	-----
Total Dissolved Solids (mg/L)	-----	-----	599	-----
Turbidity (NTU)	-----	-----	23	35
EC (mS/cm)	-----	-----	1.24	-----
SAR	4.2-5.8	-----	-----	-----
Fecal coliforms (CFU/100 mL)	4.7 x 10 ⁴ - 8.3 x 10 ⁵	-----	-----	-----
Fecal streptococci (CFU/100 mL)	110 - 3.8 x 10 ⁵	-----	-----	-----

Table 2.5 Graywater Characteristics from Various sources (Part 2 of 2)

Parameter	Source			
	Gross et al. (2005) Composite from 6 family homes (includes Kitchen)	Shafran et al. (2006) Showers, Sink, Laundry, Kitchen	Gerba et al. (1995) Shower, Washbasin, Kitchen Sink (w/o garbage disposal), Laundry	Friedler (2004) Bathtub, Shower, Laundry
pH	6.7	8.1	6.7-7.7	7.14-7.5
NH ₄ -N (mg/L)	-----	-----	-----	-----
Nitrate (mg/L)	-----	-----	1.8-3.0	0.39-4.9
Total Nitrogen (mg/L)	14.0	19	-----	-----
Sulfur (mg/L)	-----	-----	-----	-----
Sulfate (mg/L)	-----	-----	-----	-----
Boron (mg/L)	0.6	1.3	-----	0.35-0.44
Phosphorus (mg/L)	17.7	31	-----	-----
Phosphate (mg/L)	-----	-----	-----	4.56-169
Calcium (mg/L)	-----	-----	-----	-----
Chloride (mg/L)	-----	-----	-----	166-450
Magnesium (mg/L)	-----	-----	-----	-----
Potassium (mg/L)	-----	-----	-----	-----
Sodium (mg/L)	-----	-----	-----	112-530
COD (mg/L)	-----	-----	-----	230-1339
BOD (mg/L)	270	133	119.8	173-462
Total Suspended Solids (mg/L)	138	-----	19.1 - 48	78-303
Total Dissolved Solids (mg/L)	-----	-----	-----	-----
Turbidity (NTU)	-----	-----	15.3-78.6	-----
EC (mS/cm)	-----	1.7	-----	1.20-2.46
SAR	4.8	5.9	-----	-----
Fecal coliforms (CFU/100 mL)	106	9 x 10 ⁵	5.4 - 7.2 log 10	3.5 x 10 ³ - 4 x 10 ⁶

2.3.3.1 Surfactant Concentration

“Surfactants are the primary cleaning agents used in laundry and cleaning products and the highest volume surfactants currently being used in commerce are alcohol ethoxylates (AE), alkyl ethoxy sulfate (AES) and linear alkylbenzene sulfonate (LAS)” (McAvoy et al. 1998).

Surfactant concentrations vary according to the graywater source (Figure 2.1). Detergent concentrations for sulfonate based detergents in the range of 45 mg/L to 170 mg/L have been reported (Faruqui and Al-Jayyousi 2002). Using the methylene blue active substances (MBAS) method, Friedler (2004) measured anionic surfactant concentrations in graywater in the range of 3.3 mg/L to 61 mg/L. Similarly, using MBAS, Shafran et al. (2006) found that concentrations of anionic surfactants in graywater effluents were in the range of 0.7 mg/L to 70 mg/L with an average of 16.7 mg/L and were higher than anionic surfactant concentrations measured in raw domestic wastewater (0 to 10 mg/L with an average of 5.4 mg/L). Holt et al. (1998) also measured LAS concentrations in untreated domestic wastewater on average in the range of 2.52 mg/L to 4.42 mg/L.

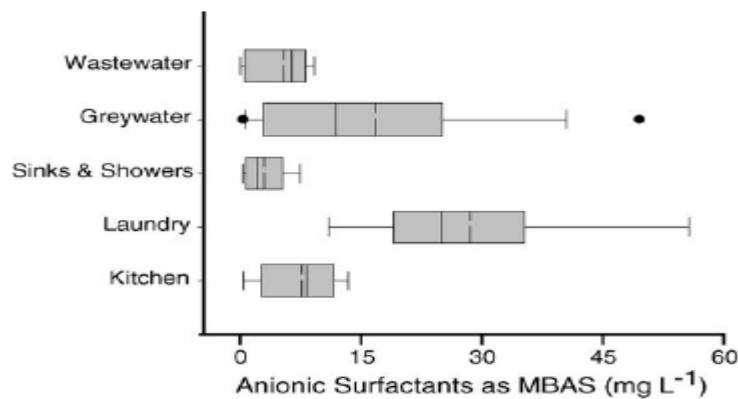


Figure 2.1 Characterization of Surfactants from Various Sources
(Shafran et al. 2006)

Gross et al. (2005) also measured anionic surfactant concentrations (by MBAS) in the range of 29-60 mg/L with an average value of 40 mg/L. Measured surfactant concentrations in treated wastewater effluent have been found to be 39 µg/L, 6.2 µg/L, 6.5 µg/L, and 5.7 µg/L for LAS, AE, AES and alcohol sulfate (AS) respectively when the influent surfactant concentrations were on average 5.2 mg/L, 3.0 mg/L, 3.2 mg/L and 0.6 mg/L for LAS, AE, AES and AS respectively (Matthijs et al. 1999).

The measured surfactant concentration will vary in a given year based on the household consumption of the surfactant. For the same sampling sites, Holt et al. (1995) measured LAS concentrations in the range of 11.8 mg/L to 18.2 mg/L (on average 15.1 mg/L) when the LAS consumption in the sampling month was 4.5 gallons per capita per day (gpcd). When the LAS consumption in the sampling month was 3.3 gpcd, Holt et al. (1998) measured LAS concentrations in the range of 2.52 mg/L to 4.42 mg/L.

McAvoy et al. (1998) found wastewater concentrations of AES, AE and LAS in the range of 0.11-2.18 mg/L, 0.69-3.68 mg/L and 1.8-6.1 mg/L respectively. In a different study, McAvoy et al. (2002) found concentrations of LAS, AES and AE to be 14.6 mg/L, 4.8 mg/L and 0.64 mg/L respectively in wastewater. Gunnar et al. (2002) states that the theoretical amount of LAS in graywater should be about 30 mg/L based on estimated surfactant use per person in a household. Surfactant concentrations were determined by the Sharvelle laboratory at three different locations – a Colorado State University (CSU) dormitory, a Fort Collins Household and at a site in Arizona. The measured AES concentrations were found to have a good correlation to values reported in literature (Table 2.6).

Table 2.6 Surfactant Concentrations

Surfactant	McAvoy et al. 2002 Septic Tank Effluent Concentration	Holt et al. 1995	Matthijs et al. 1999	Gunnar et al. 1999	¹ Arizona Household	^{1, 2} CSU Dorm	¹ Ft. Collins Household
LAS (mg/L)	14.60	15.4	5.0	30.00	1.64	1.83	0.51
AES (mg/L)	4.80	³ NR	3.2	³ NR	3.51	3.93	4.48
AE (mg/L)	0.64	³ NR	3.0	³ NR	0.00	0.46	1.57

¹ Surfactant concentrations in graywater for Arizona, CSU Dorm and Fort Collins were measured in the Sharvelle Lab

² Laundry graywater is not included in the CSU Dorm graywater

³ NR: Not Reported

2.4 Summary

Based on the above literature sources, the wide variation that exists in graywater characteristics is apparent. Difficulty often exists in making long-term predictions on the environmental effects of graywater given the unpredictability in input concentrations. With the review of the concentrations of graywater constituents conducted in addition to appropriate parameters to simulate also reviewed, the foundation was laid for the determination of the synthetic graywater recipe (Chapter 3 - Section 3.5).

Chapter 3

EXPERIMENT SETUP, MATERIALS AND METHODS

3.1 Objective

Due to difficulties that exist in determining the migration pathways and transport of contaminants in graywater by field studies, it is important to make assessments about contaminant transport under controlled conditions. Controlled conditions imply the elimination of environmental effects and variability introduced in field settings such as the effects of weather, the presence of pets in a home and the variability in graywater application volume and chemical composition. To remedy this problem of experimental variability under field conditions, a study was set up to assess the possible impacts of graywater on groundwater. Elimination of experimental variability was accomplished by setting up the experiment in a greenhouse with synthetic graywater used for the experiments. The synthetic graywater served as a way to ensure consistency and repeatability of the results. Leachate analyses were conducted for surfactants, salts, as well as other chemical parameters. Assessments were made based on the leachate analysis on the possible accumulation and subsequent leaching of graywater constituents.

It should be noted that although the experiments were conducted with the use of synthetic graywater under controlled conditions, the testing procedures adapted for the

water quality parameters measured and other constituents measured were the same as the testing procedures adopted for field tests. As previously noted, in addition to the use of synthetic graywater (and thus the elimination of the variability in contaminant loading rate), the other difference between field conditions and conditions in these experiments results from the elimination of factors (such as environmental factors and household demographics) that can serve to skew the results of measured parameters under field conditions.

3.2 Custom Plant Pot Construction

In field studies evaluating the effect of graywater application for residential landscape irrigation, the Sharvelle group performs soil sampling in intervals starting from 15 cm to 3 ft. In order to ensure adequate leaching depth and simulate field conditions, 24 in. Schedule 40 polyvinyl chloride (PVC) pipes (Kelly Supply Co., Fort Collins, CO.) were used to construct columns that encased 20 in. of soil. PVC was selected because it is readily available, relatively inexpensive and easy to use for fabrication purposes. These fabricated columns served as custom plant pots used for the duration of the experiments.

A drawback associated with PVC use is the leaching of toxins to plants from the PVC material. The harmful effect of PVC was abated by exposing the cut pipe sections to sunlight for a period of six weeks. Sunlight exposure was performed to minimize degradation of PVC and subsequent release of toxins into plant pots during the duration of the experiment. The setup of the 6 in. diameter plant pots involved a two-chamber system in which the top 22 inches encased the soil and plant biomass and the bottom 2 inches served as a drainage layer (Figure 3.1). The top chamber contained the 20 in. soil

layer with an overlying freeboard of 2 in. for the plant biomass. A 6 in. diameter perforated metal plate covered with filter fabric (Figure 3.2) was used to separate the top and bottom chambers and thus prevent soil particles from entering the drainage layer (which could lead to clogging problems). It should be noted that the drainage layer did not consist of any further filter materials, but was instead a 2 in. allotted space through which the soil leachate accumulated prior to drainage from the bottom of the plant pot.

The perforated metal plate was fitted on top of a small circular ring of PVC that was glued to the inside of the cut pipe section using PVC glue 2 inches from the bottom of the pipe. The sides of the PVC ring were sealed with silicone to prevent the passage of water and soil sediments. The circular ring served as a support for the perforated metal filter plate. The PVC pipes with the added perforated metal filter plate and PVC ring were again exposed to sunlight for 2 days. A 6 in. schedule 40 PVC cap was fitted at the bottom of the pipe. The plant pots rely on gravitational flow. Therefore, a hole was drilled at the bottom of the PVC cap to allow for leachate drainage and a hose barb adapter was screwed to the bottom of the cap (Figure 3.3). A 1/8 in. x 1/4 in. tube was attached to the hose barb adapter and was used as a means to ensure uninterrupted leachate flow from the drainage layer to the leachate storage bay.

Sample ports (though not implemented in the final design) are proposed so that soil samples from different depths can be collected and analyzed if needed. The proposed depths are 6 in. and 12 in. As of September 2010, soil sample collection has not been carried out, but will be performed after the last leachate sample is collected. The cleaning protocol for the PVC pipes, pipe caps, perforated metal plates and circular rings involved the use of water, methanol and finally deionized water.

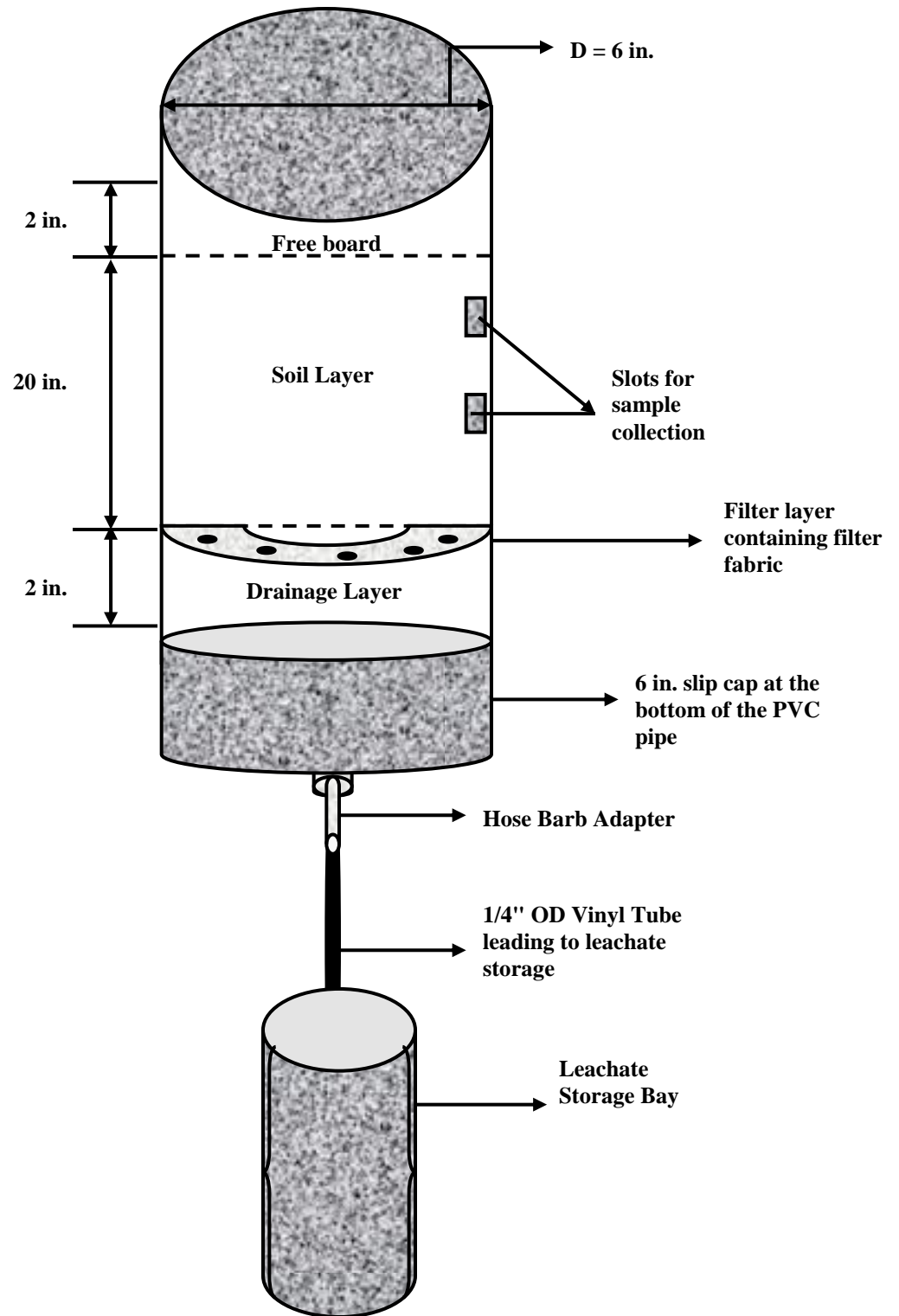


Figure 3.1 Plant Pot Schematic



Figure 3.2 *Upper Left* Metal Plate for Filter Layer; *Upper right:* Metal Plate fitted with the Filter Material; *Lower Left:* Fitted PVC Circular Ring; *Lower Right:* Top View of Filter Layer



Figure 3.3 *Upper Left:* PVC Cap with Hose Barb Adapter; *Upper right:* PVC Cap with Adapter and Tube; *Lower Left:* Support Structure for Tall Fescue Pots; *Lower Right:* Finished System of 38 Custom Plant Pots

Thirty-eight plant pots were constructed to hold the soil and plant (or grass) biomass (and in some cases only soil; Section 3.3.1) and set up in the Colorado State University (CSU) Greenhouse (Plant Growth Facilities; Figure 3.4). The supporting structure used for the plant pots with bermudagrass and the plant pots with no soil had a length of 27.5 inches, width of 18.5 inches and height of 22 inches.. For tall fescue, a longer structure with a total length of 36 inches was used. Each grid space measured 7.3 in. x 7.3 in. For the plants, given that more room is required for growth, there was additional 9 in. x 9 in. spacing between the plants for a total length of 42 inches and width of 42 inches.

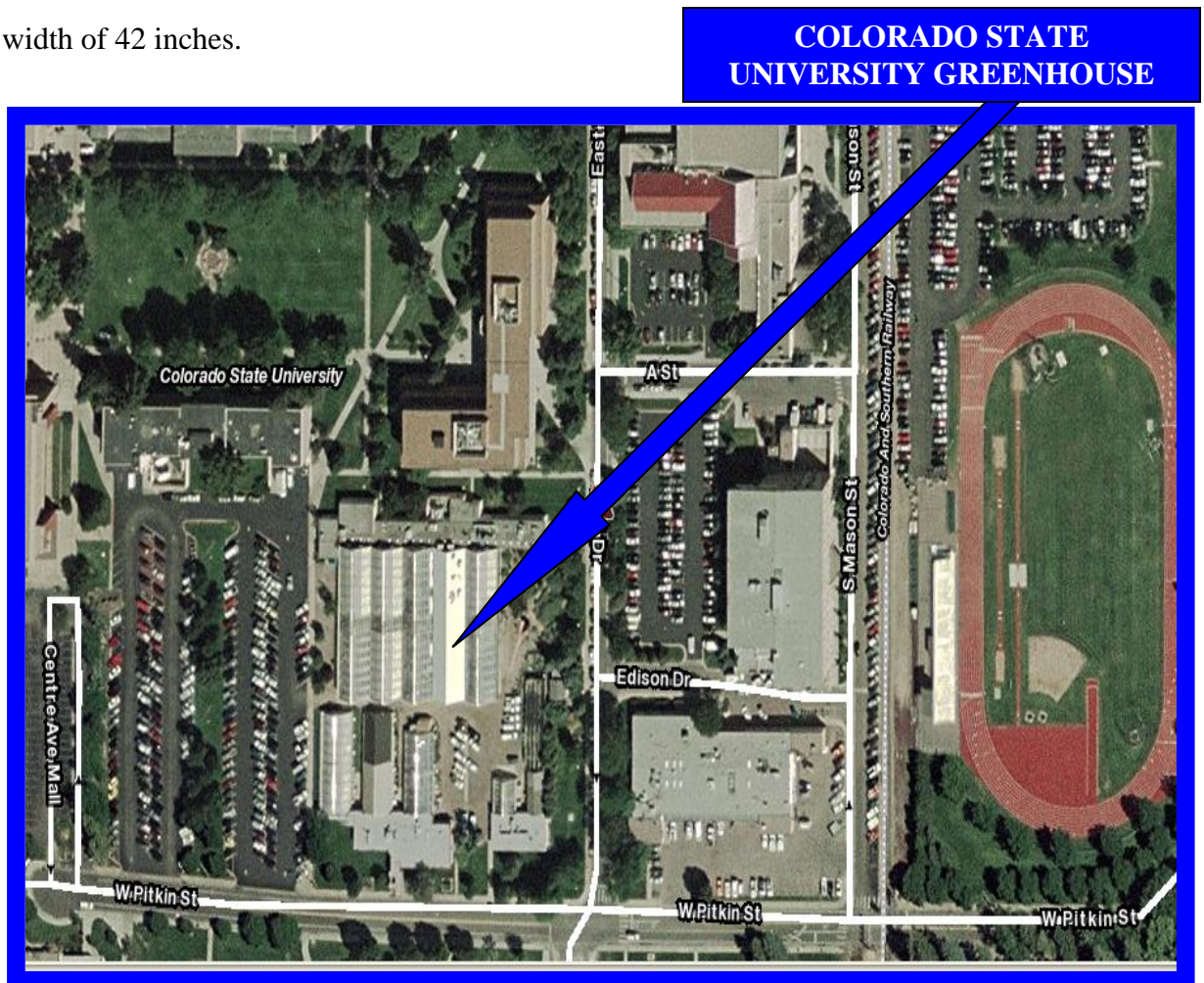


Figure 3.4 Aerial View of the Colorado State University Greenhouse

3.3 Experiment Setup

3.3.1 Plant and Grass Setup

During the course of this set of experiments, two different plant and grass types were utilized. The turfgrasses used were selected to be representative of not only turfgrasses commonly found in residential areas, but also representative of different geographic regions with varying climate. The turfgrasses used were bermudagrass (a warm season grass) and tall fescue (a cool season grass). The plants used were meyer lemon (a citrus) and emerald gaiety euonymous (a shrub). Given the long period of time required between the planting of seeds and the maturity stage for both the plants and grasses, the decision was made not to raise the plants and grasses from seeds. Instead, the plants were obtained from local greenhouses in Fort Collins, Colorado, the grasses were obtained from sod farms, and both the plants and grasses were transplanted into the custom plant pots.

The plants were severely root bound (Figure 3.5); therefore, extensive washing of the plant roots was carried out to remove as much of the greenhouse potting soil as possible before replanting. Some native greenhouse soil mix remained. However, due to the minimal depth and mass occupied by the soil mix compared to the entire depth of the soil column, the effects of the mixture of the greenhouse soil and the soil used for the experiments are expected to be negligible such that the infiltration capacity in the soil column remains unaffected. After the desired amount of soil was compacted in the plant pot, approximately 8 inches of soil was removed from the top of the compacted soil column. The base of the plant root bulb was placed on top of the soil that remained in the column, after which the removed soil was placed back gradually and carefully along the

sides of the plant back up to the top of the plant until all the excavated soil had been placed back into the custom plant pots.

For the grasses, 6-inch diameter pieces were cut from sod and placed in the plant pots on top of the compacted soil. Minimal pressure was applied to the sod to ensure that there was adequate contact between the soil and grass roots and thus guarantee that the grass roots would grow into the soil and result in the binding of the sod to the soil in the custom plant pots. A total of eight tall fescue, eight bermudagrass, eight euonymous and eight lemons were planted in the plant pots, and six pots were left without plants or grasses (containing only soil). One month after planting and irrigation with potable water, the plants and grasses did not display visual signs of distress from being transplanted (Figures 3.6. and 3.7).



Figure 3.5 *Left to Right:* Root Bound Euonymous prior to washing; Euonymous after washing; Initial planting of Tall Fescue



Figure 3.6 *Left to Right: Tall Fescue prior to trimming; Trimmed Tall Fescue; Bermudagrass*



Figure 3.7 *Left to Right: Meyer Lemon; Emerald Gaiety Euonymus; Plant Pot Setup showing all 38 Pots*

In order to ensure adequate establishment of the plants and grasses given the root stress incurred in the transplant process, immediate irrigation with synthetic graywater was not carried out. Irrigation with potable water was carried out for the first five months followed by the onset of graywater irrigation after this period of time. Irrigation with synthetic graywater began in February 2010.

For each plant and grass type, eight pots were used (Table 3.1). Of the eight pots used, four were adapted to continuous graywater irrigation (after the initial period of irrigation with potable water) and the other four were adapted to continuous potable water irrigation. Four pots for each irrigation type were included primarily for visual observation and to account for plant death unrelated to irrigation. However, leachate analysis was only performed on three of the four plant pots for each type of irrigation (synthetic graywater versus potable water) and for each plant (or grass) type. As a control, six pots without plants, containing the same soil as the pots with plant or grass biomass were included with three pots irrigated with synthetic graywater and the other three pots irrigated with potable water.

Table 3.1 Experiment Setup for Synthetic Graywater

Pot Setup	Number of pots with synthetic graywater applied	Number of pots with potable water applied
Lemon	4	4
Euonymous	4	4
Bermudagrass	4	4
Tall Fescue	4	4
Soil Only (No Plant/Grass)	3	3

3.3.2 Soil Preparation and Analysis

After the custom plant pots had been constructed and set up in the CSU greenhouse, soil was added to the plant pots. The soil used was sandy loam soil (Pioneer Sand Company, Fort Collins, CO.). The same soil (sandy loam soil) was used throughout the duration of the experiments. Although the transport of graywater constituents,

surfactants in particular, will vary with different soil types, budgetary constraints required that the experiments be conducted in one soil.

Prior to the start of the experiments, the sandy loam soil was analyzed at the Soil Water and Plant Testing Laboratory at the CSU campus in order to determine the soil properties such as the pH and background concentrations of nutrients. The soil was found to already contain nitrogen (in the form of ammonium ($\text{NH}_4\text{-N}$) and nitrate ($\text{NO}_3^- \text{-N}$)) and also contain calcium (Ca), magnesium (Mg), and sodium (Na) (Table 3.2), all of which were measured in the collected leachate. Therefore, the concentration of these constituents was taken into consideration when the results of the conducted leachate analysis were evaluated.

The soil was also found to have 65% sand, 17% silt and 18% clay. Based on the soil textural triangle (Figure 3.8), it is expected that for sandy loam soil, the percentage of sand, silt and clay should range from 50-70% sand, 15-20% clay and less than 50% silt. Therefore, the measured percentages of sand, silt and clay are in line with what is expected for sandy loam soil.

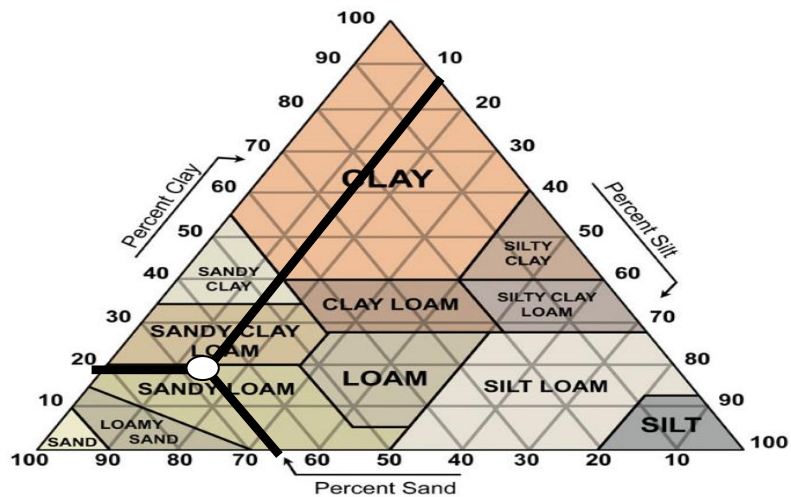


Figure 3.8 Soil Textural Triangle (*SoilSensor.com 2008*)

Table 3.2 Soil Analysis Results

Parameter	Value	Parameter	Value
pH	7.5	B (ppm)	55.4
EC (mmhos/cm)	1.3	Ca (meq/L)	5.8
Lime Estimate	Low	Mg (meq/L)	2.1
% OM	1.5	Na (meq/L)	7.1
NH₄-N	1.9	K (meq/L)	0.3
NO₃⁻-N	13.1	SAR	4.0
P (ppm)	70.0	CEC (meq/100g)	12.17
K (ppm)	91.3	Total N (%)	0.0340
Zn (ppm)	2.1	Total C (%)	0.5891
Fe (ppm)	24.6	Sand (%)	65
Mn (ppm)	2.0	Silt (%)	17
Cu (ppm)	2.9	Clay (%)	18

Cation exchange is an adsorption process that involves the displacement of an adsorbate from the soil surface by an adsorptive (Essington 2004). The cation exchange capacity (CEC) of a soil is the moles of adsorbed cation charge that can be displaced by an index ion per unit mass of soil and refers to the maximum negative surface charge (Essington 2004). In soils, the sources of cation exchange are clay minerals, organic matter and amorphous minerals (Sparks 2003).

Inorganic and organic aqueous species that exist as cations in soil solutions can undergo cation exchange reactions (Essington 2004). It should also be noted that the anion exchange capacity (AEC - the sum of the total exchangeable anions that a soil can adsorb) is important in determining the leaching potential of anions (Sparks 2003). The capacity of a soil to retain ions in a form that they are available for plant uptake and not susceptible to leaching in the soil profile is determined by the ion exchange capacity (sum

of the CEC and AEC) (Sparks 2003). In soils with substantial AEC, sulfate and nitrate can be held, but when soils lack the ability to retain anions, sulfate can be readily leached out (Sparks 2003). In subsoils that have oxides as discrete particles or as coatings on clay, the oxides impart positive charge or an AEC to the soil such that sulfate accumulation occurs (Sparks 2003).

The cation exchange capacity (CEC) of the sandy loam soil used in this study is 12 meq/100 g. The pH of the sandy loam soil was found to be 7.5. Given the low clay content of the soil (18 %), the CEC is fairly high. This high CEC can be attributed to several factors including the soil pH in conjunction with the presence of clay minerals such as kaolinite and montmorillonite. Montmorillonite has a high CEC due to the presence of fully expanded interlayers that promote exchange of ions (Sparks 2003) and can subsequently affect the soil CEC. In addition, as pH increases, deprotonation of functional groups occurs leading to greater negative charge on organic matter and clay minerals thus leading to an increase in the CEC (Sparks 2003).

It was found that there are background levels of antimicrobials and surfactants in the sandy loam soil (Tables 3.3 and 3.4). The sandy loam soil obtained from Pioneer Sand Company, Fort Collins, CO., was field soil. Therefore, background concentrations of surfactants and antimicrobials present in the soil can be attributed to exposure to these constituents that could have occurred where the soil was excavated as well as through transport and handling conditions prior to the collection of this soil and subsequent preparation of the soil for use in the experiment. The native greenhouse soil mix for the plants was also analyzed for background concentrations of surfactants and antimicrobials given that the greenhouse soil mix was present in the soil column (Section 3.3.1). No

detectable level of antimicrobials or surfactants was found for the greenhouse soil mix from the lemon plants.

Table 3.3 Soil Antimicrobial Data

	² TCS (µg/kg of soil)	³ TCC (µg/kg of soil)
Sandy Loam (1)	0.025	0.022
Sandy Loam (2)	0.029	0.027
Sandy Loam (3)	0.030	0.028
Average Sandy Loam	0.028	0.026
Lemon (1)	¹ ND	¹ ND
Lemon (2)	¹ ND	¹ ND
Average Lemon	¹ ND	¹ ND

¹ ND = Non Detectable

² TCS = Triclosan

³ TCC = Triclocarban

Table 3.4 Soil Surfactant Data

	² LAS (C10-13) (µg/kg)	³ AES (EO0-3) (µg/kg)
Sandy Loam (1)	3.3	¹ ND
Sandy Loam (2)	5.4	2.7
Sandy Loam (3)	1.9	¹ ND
Average Sandy Loam	3.53	2.7
Lemon (1)	¹ ND	¹ ND
Lemon (2)	¹ ND	¹ ND
Average Lemon	¹ ND	¹ ND
Euonymous (1)	2.2	1.8
Euonymous (2)	¹ ND	¹ ND
Average Euonymous	2.2	1.8

¹ ND = Non Detectable

² LAS = Linear alkylbenzene sulfonate

³ AES = Alkyl ethoxy sulfate

The soil used in this study was compacted to a bulk density of 1.5 g/cm^3 . Compaction of the soil was carried out using a 1-inch dowel rod (Figure 3.9). A bulk density of 1.5 g/cm^3 is typically used in lab scale column studies. Abu-Zreig et al. (2003) obtained a bulk density of 1.49 g/cm^3 for Caledon sandy loam soil by compaction in capillary rise experiments. Prior to the start of irrigation with synthetic graywater, methods for determination of quantity and frequency of irrigation events were tested and the chemical makeup of the synthetic graywater was determined.



Figure 3.9 Soil Compaction with a Dowel Rod

3.4 Determination of Evapotranspiration Rates and Irrigation Volume

Irrigation water was applied manually. The amount of water to be applied was determined based on the evapotranspiration (ET) rate and plant (or grass) type. To estimate the ET rate in the greenhouse and subsequently determine irrigation scheduling, a black bellani plate atmometer was used. A bellani plate is a thin porous blackened

ceramic disc about 2.95 inches in diameter fused to the large end of a glazed ceramic funnel. The initial planned set up for the black bellani plate system is illustrated in Figure 3.10.

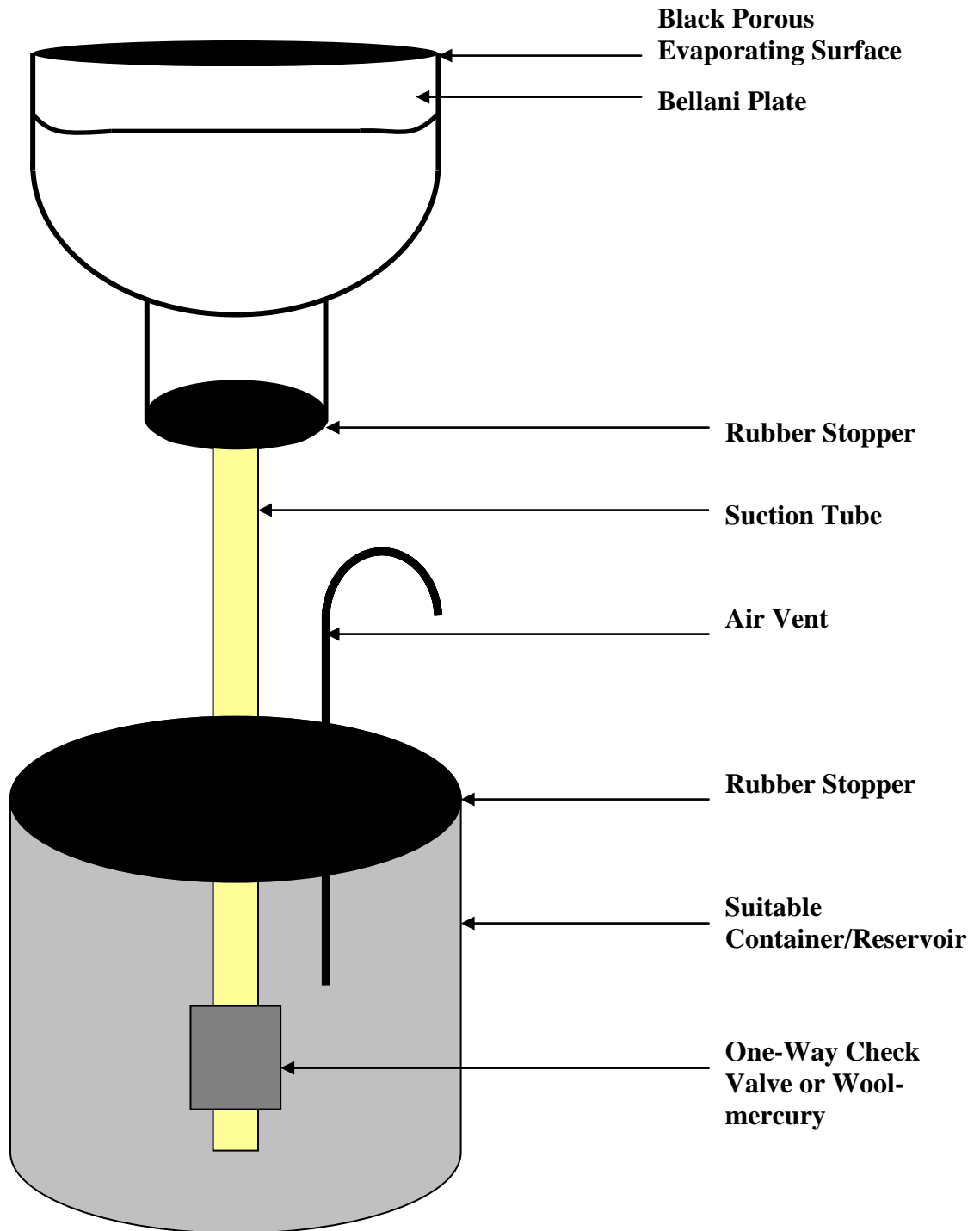


Figure 3.10 Black Bellani Plate Setup

Some of the advantages of the black bellani plate atmometer include that it is small, simple, inexpensive, easy to setup, operate and maintain, and the evaporating surface cannot be deformed (Robertson and Holmes 1957). The black bellani plate was selected not only because of the advantages it provides, but because it has been successfully adapted in field conditions for the measurement of evaporation by researchers.

Initially, the black bellani plate was suspended above an Erlenmeyer flask filled with water and connected via a tube. However, minimal evaporation was observed, and there were also suction problems encountered with the transport of water to the black bellani plate from the Erlenmeyer flask. Therefore, another setup was adopted (Figure 3.11) based on Robertson and Holmes (1957).



Figure 3.11 *Left to Right:* Black Bellani Plate – use of an Erlenmeyer flask; Black Bellani Plate – use of a Burette (top Section); Black Bellani Plate – use of a Burette (bottom Section)

Robertson and Holmes (1957) used a black bellani plate to determine ET. However, instead of suspending the bellani plate above a reservoir of water, a burette of water was used and the connection between the burette and bellani plate was in the form of a U-shaped tube. The burette serves as both a reservoir and measuring device. The black bellani plate was used to measure the latent evaporation. The Robertson and Holmes (1957) setup was adopted and the results obtained for the daily evaporation were consistent and compared closely to the evaporation from an open beaker (setup in the greenhouse). These latent evaporation results were incorporated in the determination of the ET. The black bellani plate was suspended 6 inches above the ground surface.

From Robertson and Holmes (1957), pan evaporation (in inches) is related to latent evaporation (in cubic centimeters) by the equation $ET_{pan} = 0.03 + 0.0032LatentE_{bellani}$ (Figure 3.12). The value of 0.03 which is the y intercept from the correlation relationship between pan evaporation and latent evaporation can be considered insignificant such that the relationship between the pan evaporation (P.E.) and latent evaporation (L.E.) is 0.0032 inches of P.E. per cubic centimeter (c.c) of L.E. This relationship of P.E. to L.E. is similar to that reported by Carder (1968) in which the water evaporated is calculated as the mL of water lost from the surface of the bellani plate multiplied by 0.0028.

The ET from the plant (or grass) can be calculated as $ET_{plant} = coef. \times ET_{pan}$ with the appropriate plant (or grass) coefficient (coef.) applied. For bermudagrass, coef. = 0.8 to 0.85 for tall fescue, coef. = 0.9 to 0.95, for citrus, the crop coefficient is 0.45 to 0.55 (no ground cover and 20% canopy) and for shrubs, a wide variation of crop coefficient exists with a variation of 0.3 to 0.9 (Allen et al. 1998).

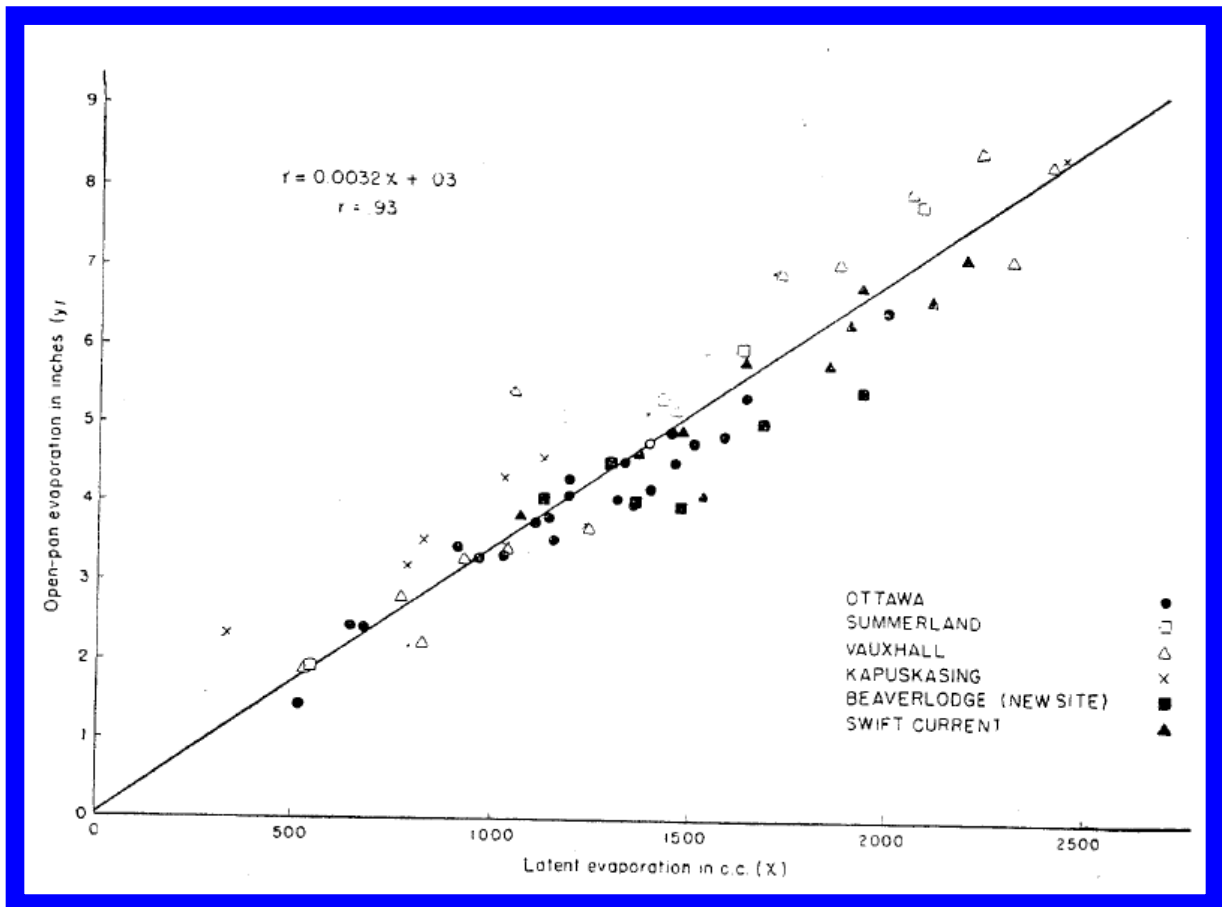


Figure 3.12 Relationship between monthly open pan evaporation and monthly latent evaporation (*Robertson and Holmes 1957*)

The latent evaporation in the CSU greenhouse was determined to be approximately 20 mL per day using the black bellani plate atmometer. This latent evaporation value was set as the baseline latent evaporation, and subsequent start point for the ET computation. This latent evaporation value of 20 mL was also compared to the evaporation from a small beaker setup in the CSU greenhouse next to the black bellani plate and similar in diameter to the black bellani plate. The evaporation from the beaker was 25 mL per day. From the value obtained from the black bellani plate, the ET for each grass and plant was

determined using the relationship between P.E. and L.E. provided by Robertson and Holmes (1957) and the appropriate crop coefficient. However, the latent evaporation can undergo seasonal variations. Therefore, the latent evaporation from the black bellani plate atmometer was monitored in order to make adjustments to the irrigation volume if necessary. The irrigation amount for each plant and grass was set as 120% of the plant (or grass) ET to ensure leachate generation (Table 3.5).

Table 3.5 Determination of ET

	Crop Coeff.	Volume of ET (in³)	Volume of ET (cm³)	Crop ET (mL)	Irrigation Volume (L) = 120% ET	Irrigation Amount (in) = 120% ET
Euonymous	0.60	113.41	1858.48	1115.09	1.34	2.89
Lemon	0.55	113.41	1858.48	1022.16	1.23	2.65
Tall Fescue	0.95	113.41	1858.48	1765.56	2.12	4.56
Bermudagrass	0.85	113.41	1858.48	1579.71	1.90	4.10

Prior to the start of irrigation with synthetic graywater, it was observed that in some cases leachate was not being generated from some of the pots containing euonymous and lemon. After close monitoring of this leachate generation problem and investigation of the drainage layer of the plant pots for possible clogging, the decision was reached to increase the irrigation volume for the euonymous and lemon. This decision was reached given that no clogging problems were discovered and due to the fact that the canopy cover for the plants was substantially larger than the diameter of the plant pots.

The soil moisture was measured (Table 3.6) and it was discovered that even at irrigation at a 120 % ET, the plants were not being over watered. The soil moisture

provided is an average for the eight euonymous, lemon, bermudagrass and tall fescue pots with measurement of the soil moisture for each plant pot carried out immediately after irrigation.

Table 3.6 Soil Moisture Data

	Soil Moisture (%)
Euonymous	22.5
Lemon	26.4
Tall Fescue	38.0
Bermudagrass	29.0

It was observed that the soil moisture for bermudagrass and tall fescue was higher than that for euonymous and lemon. To make the adjustment for the irrigation volume for the euonymous and lemon, the maximum provided crop coefficients for these plants were utilized instead of the previous mid range value used. Therefore, the new crop coefficients for euonymous and lemon adopted were respectively 0.90 and 0.65 with weekly irrigation volumes for the euonymous and lemon of 2.01 L (4.34 in.) and 1.45 L (3.13 in.) respectively. These amended volumes were applied for the determination of the fertilizer to be added to the plants and grasses (Section 3.6). There was substantial leachate generation from the plant pots containing bermudagrass such that the weekly applied irrigation amount was decreased. The plant pots with only soil were irrigated with the same irrigation volume as the euonymous.

3.5 Synthetic Graywater

The synthetic graywater was formulated to contain constituents typically found in actual graywater including nutrients. Because boron is often found in Western state soils and potentially graywater, and can have toxic effects on plant life depending on the concentration, boron was also included in the graywater composition. During this study, comparisons had to be made between the leachate analysis results from graywater and potable water irrigated pots. To that effect, the nutrient levels in the synthetic graywater were not synthesized to be the same as the nutrient levels in the potable water. The composite graywater composition was determined based on literature values (Chapter 1 – Table 1.2, Chapter 2 – Tables 2.4, 2.5 and 2.6). The synthetic graywater was formulated based on predetermined desired concentrations of various constituents as well as established water quality parameters from these literature sources.

In cases where a concentration range was provided in the literature sources, the average over this range was determined. From these average values, the overall average for each constituent was determined. The average value in most cases was used as the composite concentration. Exceptions were made in cases where the average value was skewed due to an unusual high or low value for a particular constituent (adjustments were made for sodium and boron). The values for sodium and magnesium were adjusted to maintain the SAR below 5.0. As a method of comparison to literature sources, measured calcium, magnesium and sodium concentrations along with the calculated SAR determined by the Sharvelle laboratory from a CSU residence hall (dormitory) and an Arizona household (AZ new installation home, Phoenix) have been included (Table 3.7). The final composite concentration (Table 3.8) was determined based on the

literature values (Tables 1.2, 2.4, and 2.5) and the values measured by the Sharvelle laboratory (Tables 2.6 and 3.7). Included in Table 3.8 are also the minimum and maximum values based on Tables 1.2, 2.4, 2.5, and 3.7. It should be noted that the SAR provided in Table 3.8 for the composite synthetic graywater concentration is the calculated SAR based on the concentrations of calcium, magnesium and sodium used for the synthetic graywater, not the average SAR determined from the literature sources. Some of the reviewed literature included kitchen wastewater. Measured graywater constituents and parameters that included kitchen wastewater (although provided in Table 2.5), were not used for the determination of the final composite values used for the synthetic graywater composition.

Linear alkylbenzene sulfonate (LAS), alkyl ethoxy sulfate (AES) and alcohol ethoxylate (AE) were also included in the synthetic graywater. These surfactant concentrations were determined based on the LAS, AES and AE concentrations provided in Table 2.6 in addition to other literature sources. The concentrations of LAS, AES and AE used in the synthetic graywater were 21.0 mg/L, 4.5 mg/L and 0.9 mg/L respectively.

Table 3.7 SAR for CSU Dormitory and Arizona Household

	Ca (mg/L)	Mg (mg/L)	Na (mg/L)	SAR
Arizona	8.9	4.9	35.0	2.34
Dorm	4.0	0.43	2.7	0.34

Table 3.8 Synthetic Graywater Composition

Concentration	Minimum	Maximum	Composite
Temperature (°C)	21.6	28.2	24.90
pH	6.54	8.1	7.35
COD (mg/L)	144	657.5	378.20
BOD (mg/L)	39	119.80	75.41
TSS (mg/L)	35.09	107	71.05
Turbidity (NTU)	35	130	66.25
NH ₄ -N (mg/L as N)	0.22	6.13	2.22
NO ₃ -N (mg/L as N)	0.14	3.90	1.66
¹ Total N (mg/L)	1.7	7.6	¹ 3.88
PO ₄ -P (mg/L as P)	0.105	9.3	0.80
² Total P (mg/L)	0.53	51.32	² 0.80
Sulfate (mg/L)	22.9	59.59	45.88
Chloride (mg/L)	9	20.54	14.77
Hardness (mg/L as CaCO ₃)	141.50	144	142.75
Alkalinity (mg/L as CaCO ₃)	158	158	158
Ca (mg/L)	7.95	99.5	54.93
K (mg/L)	2.35	9.05	6.99
⁵ Mg (mg/L)	2.0	46.6	⁵ 11.47
³ Na (mg/L)	23.5	340.5	³ 26.00
⁴ B (mg/L)	0.3	1.37	⁴ 0.50
TDS (mg/L)	559	660	609.50
EC (µS/cm)	795	1309.50	1052.25
⁶ SAR	0.84	5.9	⁶ 0.83

¹ The Total N was calculated as the sum of NH₄-N and NO₃-N (not based on average source values)

² The Total P was determined as the value of just PO₄-P, the only source contributing P (not based on the overall average from literature sources)

³ Average value for Na is 149.27 mg/L, but taking into account the high range of sodium and to regulate the SAR, a value of 28.00 mg/L was instead used

⁴ Average value for B is 0.835 mg/L, but boron levels above 0.5 mg/L can be harmful to plants, a value for B of 0.50 mg/L is used for the synthetic graywater

⁵ Average value for Mg is 16.95 mg/L

⁶ SAR calculated based on composite concentrations of Na, Ca and Mg (not based on the overall average)

The surfactants added to the graywater were obtained from Shell Chemicals and Stepan. Yeast extract was added to account for additional carbon content typical in graywater beyond the contribution from surfactants. The chemical oxygen demand (COD) for the surfactants and a sample of yeast extract were measured (Table 3.9) in order to determine the amount of yeast extract to add to the graywater (Table 3.10) in order to achieve the target COD concentration of 378 mg/L. Analysis of field soils and water samples collected and analyzed in the Sharvelle laboratory revealed that AES (E2) and AES (E3) are more dominant in commercial detergents. The synthetic graywater therefore consists of AS (E0): 15-20%, AES (E1): 15-20%, AES (E2): 35-40%, and AES (E3): 25-30%.

The synthetic graywater recipe (Table 3.11) was developed based on the target graywater composition (Table 3.8 and the surfactant concentrations provided above). In developing this graywater composition, it was found that in order to achieve the desired concentration of ions such as calcium and sodium by using salts, the resulting effect was that the concentrations of sulfate, chloride and nitrate were in excess. Nitrate levels were however maintained below 5 mg/L to ensure plant health. Given that even at the higher sulfate concentrations (less than 500 mg/L), it is not expected that toxic effects to plants will occur (Chapter 1 – Section 1.4.9), the sulfate was added in a substantially greater quantity relative to the target composition in comparison to other constituents like nitrate and chloride. After the necessary adjustments were made, the graywater composition was obtained with noted levels of higher nitrate, chloride and sulfate compared to the target graywater composition (Table 3.12).

Table 3.9 Measured COD for Surfactants and Yeast Extract

Commercial Surfactant Product	Class	Number of EO Groups	Components				² Average COD (mg COD/mg Component)
			Water (%)	Alkyl (C10-C16) ether sulfate, sodium salt (%)	Other		
Stepanol DCFAS-N	AS	0	3.0-5.0 (*3.8)	Sulfuric acid, mono C12-C18-alkyl esters, sodium salts > 90 (*92.1)	Alcohols, C12-18 (%)	< 4 (*1.9)	0.44
Steol CS-130	AES	1	72-76	24-26 (25.19)	Alcohols, C12-14, ethoxylated (%)	< 2	0.54
Steol CS-270	AES	2	24-32	68-72	Polyethylene glycol monododecyl ether (%)	0-2.5	1.52
Steol CS-330	AES	3	67-75	Sodium Laureth (n => 3) sulfate 27-30 (*27.93)	Alcohols (C12-15 Ln. saturated) ethoxylate (%)	0-2	0.62
Neodol 25-9	AE	¹ NA	0.0	C12-C15 Alcohol Ethoxylate 100.0% W	Ethylene Oxide (ppm)	<6.00	2.95
Bio-soft D-40	LAS	¹ NA	60-62	Sodium dodecylbenzenesulfonate 34-36 (*37.88)	Sodium xylenesulfonate (%)	2-4	0.95
Yeast Extract	¹ NA	¹ NA	¹ NA	¹ NA	¹ NA	¹ NA	1.27

¹ NA: Not Applicable

² Calculated Average COD – correction not made for percentage active

*() Value in parenthesis represents analysis result for that surfactant as reported in the surfactant data sheet

Table 3.10 Determination of Yeast Extract Concentration

Surfactant	Desired Composite Concentration		% Active	Concentration of Added Surfactant Product Based on % Active (mg/L)	Average COD (mg COD/mg Component)	COD Contributed (mg/L)
AES	Total = 4.50 mg/L		¹ NA	¹ NA	¹ NA	¹ NA
Steol CS-130	15% Contribution	0.675 mg/L	25.19	2.68	0.54	1.45
Steol CS-270	15% Contribution	0.675 mg/L	70.00	0.96	1.52	1.46
Steol CS-330	40% Contribution	1.80 mg/L	27.93	6.44	0.62	3.99
Stepanol DCFAS-N	30% Contribution	1.35 mg/L	92.10	1.47	0.44	0.65
AE	Total = 0.90 mg/L		¹ NA	¹ NA	¹ NA	¹ NA
Neodol 25-9	100% Contribution	0.90 mg/L	100	0.90	2.95	2.66
LAS	Total = 21.0 mg/L		¹ NA	¹ NA	¹ NA	¹ NA
Biosoft D-40	100% Contribution	21.0 mg/L	37.88	55.44	0.95	52.69
Total COD from Surfactants						<u>62.90</u>
COD Required to fulfill COD Composite in Synthetic Graywater (Subsequent contribution from Yeast Extract)						<u>315.30</u>

¹ NA: Not Applicable

Table 3.11 Synthetic Graywater Recipe

Reagent	Concentration (mg/L)
Ammonium Chloride <i>crystalline</i> (NH₄Cl)	8.48
Sodium Nitrate (NaNO₃)	15.78
Sodium Borate (Na₂B₄O₇·10H₂O)	4.41
Potassium Phosphate monobasic (KH₂PO₄)	3.51
Magnesium Sulfate Anhydrous <i>powder</i> (MgSO₄)	57.53
Potassium Chloride <i>crystalline</i> (KCl)	11.44
Calcium Chloride (CaCl₂·2H₂O)	47.11
Sodium chloride (NaCl)	25.61
Calcium Sulfate (CaSO₄)	143.32
Sodium Sulfate (Na₂SO₄)	40.51
Biosoft D-40	55.44
Neodol 25-9	0.90
Steol CS-130	2.68
Steol CS-270	0.96
Steol CS-330	6.44
Stepanol DCFAS-N	1.47
Yeast Extract	248.27

Table 3.12 Concentrations of the Various Graywater Constituents

	Target Composition (mg/L)	Actual Composition (mg/L)	Contributing Reagent	
NH ₄	2.22 as N	2.22 as N	8.482 mg/L NH ₄ Cl =	2.22 mg/L NH ₄ -N
NO ₃	1.66 as N	2.599 as N	15.779 mg/L NaNO ₃ =	2.599 mg/L NO ₃ -N
B	0.5	0.5	4.414 mg/L Na ₂ B ₄ O ₇ .10H ₂ O =	0.500 mg/L B
PO ₄	0.800 as P	0.800 as P	3.513 mg/L KH ₂ PO ₄ =	0.800 mg/L PO ₄ -P
SO ₄	45.88	174.321	57.527 mg/L MgSO ₄ = 40.506 mg/L Na ₂ SO ₄ = 143.321 mg/L CaSO ₄ =	45.88 mg/L SO ₄ 27.377 mg/L SO ₄ 101.064 mg/L SO ₄
K	6.99	6.99	3.513 mg/L KH ₂ PO ₄ = 11.437 mg/L KCl =	1.007 mg/L K 5.983 mg/L K
Cl	14.77	49.389	8.482 mg/L NH ₄ Cl = 11.437 mg/L KCl = 25.612 mg/L NaCl = 47.114 mg/L CaCl ₂ .2H ₂ O =	5.629 mg/L Cl 5.446 mg/L Cl 15.558 mg/L Cl 22.756 mg/L Cl
Na	28	28	4.414 mg/L Na ₂ B ₄ O ₇ .10H ₂ O = 15.779 mg/L NaNO ₃ = 40.506 mg/L Na ₂ SO ₄ = 25.612 mg/L NaCl =	0.532 mg/L Na 4.27 mg/L Na 13.118 mg/L Na 10.08 mg/L Na
Ca	54.93	54.93	47.114 mg/L CaCl ₂ .2H ₂ O = 143.321 mg/L CaSO ₄ =	12.82 mg/L Ca 42.11 mg/L Ca
Mg	11.47	11.47	57.527 mg/L MgSO ₄ =	11.47 mg/L Mg

3.6 Fertilizer Addition

A component of ensuring plant health includes the addition of fertilizer. At the onset of the experiments, it was determined that one pound of nitrogen (N) fertilizer per thousand square feet would be applied (0.089 g N for each plant pot) to both the plants and grasses treated with synthetic graywater and potable water. However, given that there is additional nitrogen added from the synthetic graywater (Table 3.12), the additional nitrogen contributed from the graywater over a four month duration was determined. Thus, the required fertilizer addition for the graywater irrigated pots was adjusted such that a smaller amount of fertilizer was added to the graywater irrigated pots compared to the potable water irrigated pots (Table 3.13).

Corrections were made for the amount of fertilizer to be added based on the percentage of nitrogen in the fertilizer applied. Osmocote slow release fertilizer that feeds up to four months was used and has 19 % total nitrogen. For the case of the potable water irrigated pots, with the correction for the percentage of nitrogen present in the fertilizer, the amount of fertilizer applied for all the plants and grasses was 0.468 g.

Table 3.13 Excess N in Graywater and Required Contribution of Nitrogen Fertilizer

	Target (mg/L)	Actual (mg/L)	Excess (mg/L)	Irrigation Amount (L)	Irrigation Amount in 4 months (L)	Excess g of N	Required Fertilizer (g of N)	Actual Fertilizer (g)
Euonymous								
NH ₄ -N	2.22	2.22	0	2.01	32.16	0.0000	0.0588	0.3095
NO ₃ -N	1.66	2.599	0.939	2.01	32.16	0.0302		
TN	3.88	4.819	0.939	2.01	32.16	0.0302		
Lemon								
NH ₄ -N	2.22	2.22	0	1.45	23.2	0.0000	0.0672	0.3538
NO ₃ -N	1.66	2.599	0.939	1.45	23.2	0.0218		
TN	3.88	4.819	0.939	1.45	23.2	0.0218		
Tall Fescue								
NH ₄ -N	2.22	2.22	0	2.12	33.92	0.0000	0.0571	0.3008
NO ₃ -N	1.66	2.599	0.939	2.12	33.92	0.0319		
TN	3.88	4.819	0.939	2.12	33.92	0.0319		
Bermudagrass								
NH ₄ -N	2.22	2.22	0	1.90	30.4	0.0000	0.0605	0.3182
NO ₃ -N	1.66	2.599	0.939	1.90	30.4	0.0285		
TN	3.88	4.819	0.939	1.90	30.4	0.0285		

3.7 Leachate Sample Collection

Throughout the duration of the experiment, the total volume of leachate generated was measured and recorded. However, leachate samples were only collected for analysis in April, June and August. For sampling and analysis, leachate samples were collected immediately after irrigation in sampling bottles. At least 300 mL for each sample was collected. However, the sample volume was dependent on the amount of leachate generated.

In order to assess the leachate quality, for each sampling event, several water quality parameters were measured. Parameters measured include total organic carbon (TOC), total nitrogen (TN), nitrate, ammonium, boron, sodium adsorption ratio (SAR), total sulfur, conductivity, linear alkyl benzene sulfonate (LAS), alkyl ethoxy sulfate (AES), alkyl sulfate (AS), alcohol ethoxylate (AE), total dissolved solids (TDS), total suspended solids (TSS), and volatile suspended solids (VSS).

In order to assess the percentage of a particular constituent that was not leached out through the soil column of the graywater irrigated pots, the percentage retained was determined. It should however be noted that the percentage retained implies that the graywater constituent could have been either held in the soil column (sorbed to soil), taken up by plants, biodegraded (in the case of surfactants) or could have undergone a combination of these three mechanisms. This percentage retained calculation was performed based on the input volumetric concentration of the particular constituent in the synthetic graywater using Equation (1) below where C_L is the concentration of the graywater constituent measured in the leachate, and C_G is the original concentration of

the graywater constituent in the synthetic graywater.

$$\text{Percentage Retained} = 100\% - \left(\frac{C_L}{C_G} \times 100\% \right) \quad \text{Equation (1)}$$

After the leachate samples were collected, within three hours, the unfiltered samples were analyzed for the conductivity, TOC and TN. After these tests were carried out, determination of the TSS, TDS and VSS was performed. After these analyses were conducted, on the same day that the sampling event occurred, the remaining samples were filtered and stored in a refrigerator. The remaining analyses were performed within 1 to 2 days. It should be noted that for the surfactants, though the sample extraction (Section 3.8.2.2) was performed within the specified period, measurement of the surfactant concentration did not occur immediately. The surfactant samples were stored in a freezer (-6°C) after extraction was complete until the samples were analyzed.

3.8 Testing Methods

The testing procedures adopted for the measurement of the required leachate analysis parameters in addition to the procedures followed for the determination of background soil concentrations and the required analyses necessary to determine the synthetic graywater composition are outlined below.

3.8.1 Organics

3.8.1.1 Chemical Oxygen Demand (COD)

For COD measurement, 2 mL of the sample was digested in COD reagent vials along with 2 mL of deionized water as the blank. The samples were heated at 150°C for two hours after which they were allowed to first cool to 120°C in the heating chamber

and then to room temperature. The samples were then removed from the heating chamber. Following this, the COD of the sample was measured using the Hach DR/2500 spectrophotometer (Figure 3.13) after first zeroing the instrument with the blank.



Figure 3.13 Hach DR/2500 spectrophotometer

3.8.1.2 Total Organic Carbon (referenced in part from *Standard Methods, 2005(5310 B)*)

There are two main methods by which TOC is measured. The two methods differ with how organic carbon is oxidized:

- (a) High Temperature Combustion Method (used in these analyses)
- (b) Persulfate ($S_2O_8^{2-}$) Oxidation Method.

The TOC was measured using TOC-VCSH Shimadzu organic carbon analyzer (Figure 3.14). The instrument relies on the oxidation of the organic carbon. Non-purgable organic carbon can be used to analyze the organic portion of carbon. The inorganic content is first removed by acidification (conversion of inorganic carbon (IC) to H_2CO_3 or CO_2) and subsequent degassing. Catalytic oxidation (combustion) is used to convert the organic content to CO_2 and this gas can be quantified using non dispersive infrared (NDIR).

Sparging is carried out to eliminate the interference caused by CO₂ released from inorganic carbon (bicarbonate). The pH is taken to about 2 to 4, and air is bubbled through, thus removing the inorganic carbon. Once the inorganic carbon has been removed, oxidation and subsequent quantification of TOC can thus be carried out.



Figure 3.14 Shimadzu Instrument

3.8.2 Surfactants

Prior to surfactant analysis, extraction of the surfactants from the samples had to be performed. A separate procedure was followed for extraction from soil samples and liquid samples. However, for final analysis of the surfactants extracted from the liquid and soil samples using liquid chromatography/mass spectrometry (LC-MS), the testing process was the same.

3.8.2.1 Surfactant Ware Protocol

Given the ease of contamination from glassware during surfactant extraction, a cleaning protocol was adopted. All surfactant glassware was cleaned by rinsing with hot water (at approximately 55°C), deionized water, and methanol. The glassware was then heated at 110-120°C for at least 1 hour before it was used. To avoid contamination, the glassware was also not placed in contact with bare skin, latex gloves, or paper products (because of the possibility of the presence of surfactants).

3.8.2.2 Liquid Extraction

Extraction for surfactants from the water samples was carried out using solid phase extraction (SPE). The extraction process involved the use of Waters OASIS extraction cartridges with an SPE vacuum (Figure 3.15). The pressure was kept at or below 5 in. of mercury (Hg). These extraction cartridges contain a unique sorbent that have a hydrophilic-Lipophilic Balance [HLB]. To begin, the Oasis HLB extraction cartridges were placed on the vacuum manifold and the vacuum was set to 5 in. Hg. A continuous vacuum was maintained on all cartridges throughout the extraction process. The cartridges were first conditioned by adding and drawing through the cartridge 1 mL of methanol. To equilibrate the system, 1 mL of water was added and drawn through the tubes. A sample volume of 1 mL was then added and drawn through the cartridges. To wash out the cartridges, 1 mL of 5% methanol in water (v/v) was added and drawn through the cartridges. The vacuum was released, the manifold cover was removed and the waste fluids were discarded. The rack containing the collection vessels was inserted into the vacuum manifold. The cover was replaced and the vacuum was turned on. To

elute, 1 mL of methanol was added and drawn through with the eluates (samples) collected and analyzed using LC-MS.



Figure 3.15 SPE Vacuum and Extraction Cartridges

3.8.2.3 Soil Extraction

In order to extract the surfactants from soil, 30 g of the soil was weighed and transferred to 50 mL centrifuge tubes. 20 mL methanol was then added to the soil contained in the centrifuge tubes by using a pipette. Automated shaking was carried out for 20 minutes at 350 rpm (Figure 3.16) followed by placement of the centrifuge tubes in a sonicator (in which water was added to heat it up) for 5 minutes. The samples were then centrifuged for 10 minutes at 2500 rpm. After centrifugation, the clear solvent was decanted to a separate centrifuge tube, and the tube was capped. The methanol extraction was repeated once with the same soil sample and additional methanol added to the first extract – that is methanol was added, automatic shaking, sonication, centrifugation and decanting of liquid were repeated. At the end of the process, approximately 40 mL of liquid was contained in the centrifuge tube. The tube was then placed under nitrogen in order to evaporate the methanol extract. Following drying, 1 mL of methanol was added

to the tube. This addition was followed by centrifugation for 5 minutes. After centrifugation, the samples were filtered using 0.45 μm sterile cellulose acetate membrane centrifuge filters. After filtration was complete, the liquid was transferred to 2 mL autosampler vials for LC-MS analysis. These vials were stored in the freezer (-6°C) prior to surfactant measurement using the LC-MS machine.



Figure 3.16 Extraction Process

3.8.2.4 Measurement

To quantify LAS, an Agilent 1200 (Agilent Technologies) High-throughput HPLC system coupled with an Agilent 6220 Accurate Mass Time of Flight mass spectrometer (Agilent Technologies) was used. Chromatographic separation was carried out with an XTerra[®] MS C18 column (2.5 μm , 50 \times 2.10 mm) from Waters. The data was controlled and processed using MassHunter Workstation software. Sample volumes of 10 μL were introduced to the instrument with an auto-sampler. The column temperature was

maintained at 40°C at both the right and left sides. A gradient method with a mixture of water with 10 mM ammonium acetate (A) and acetonitrile with 10 mM ammonium acetate (B) was used. The gradient method included an initial mix of 62% water and 38% acetonitrile. This was followed by a linear increase to 65% acetonitrile within 25 minutes, a linear increase to 80% acetonitrile for 10 minutes, with a post run at initial conditions for 10 minutes. A flow rate of 0.32 mL/min was used. To determine C₁₀₋₁₄ LAS, an electrospray ionization (negative ion mode) mass spectrometer was used. The LAS materials are C₁₀ = m/z 297, C₁₁ = m/z 311, C₁₂ = m/z 325, C₁₃ = m/z 339, C₁₄ = m/z 353.

To measure AES, an Agilent LC-MS system consisting of an Agilent 1200 high-throughput HPLC coupled with an Agilent 6220 Accurate Mass Time of Flight mass spectrometer was used. Chromatographic separation was performed using an XTerra[®] MS C18 (2.1×50 mm), 2.5 μm column. The mobile phase used for the AES analysis was: (A) Water with 10 mM ammonium acetate, and (B) Acetonitrile with 10 mM ammonium acetate. The gradient method was 38% acetonitrile and 62% water at the initial condition with a subsequent increase to 65% over 25 minutes and to 80% acetonitrile for 10 minutes with the final post run of 10 min at the initial condition. The flow rate was 0.32 mL min⁻¹ at 40°C in negative ionization mode.

For the measurement of AE, chromatographic separation was carried out with an Allure C18 (150 × 2.1 mm) and a 5 μm Restek column. A methanol–water gradient (both solvents contain 5 mM ammonium acetate to obtain the ammonium adduct) was used with a mobile phase flow of 0.32 ml/min. The gradient starts with 60% methanol during the initial 10 minutes followed by an increase in methanol to 95% for 15 minutes. This gradient was maintained for 8 more minutes. Detection was carried out using an Agilent

6220 Accurate Mass Time of Flight mass spectrometer (Agilent Technologies) with an APCI source. The conditions of the APCI are: a source in positive ionization mode, capillary voltage of 4000 V, fragmentor voltage 140 V and an APCI temperature of 400°C.

3.8.3 Antimicrobials

3.8.3.1 Soil Extraction

A similar procedure to that used for surfactant extraction from soils (3.8.2.3) was adopted for antimicrobial extraction from soils. The difference however for antimicrobial extraction was that 25 mL of 50:50 (by volume) methanol/acetone was added to the soil instead of 20 mL of methanol. In contrast to the surfactant soil extraction process, prior to the use of the shake table, the soil-methanol/acetone solution was hand shaken for 5 minutes.

3.8.3.2 Measurement

Triclosan > 97% (HPLC) and triclocarban 99% were obtained from Sigma-Aldrich. An Agilent 1200 (Agilent Technologies) high-throughput HPLC system coupled with Agilent 6220 Accurate Mass Time of Flight mass spectrometer (Agilent Technologies) was used for the determination of triclocarban and triclosan. Chromatographic separation was carried out using an Allure C18 column (5 µm, 150×2.00 mm) from Restek. The data were controlled and processed using MassHunter Workstation software. Sample volumes of 5 µL were introduced to the instrument with an autosampler. The column temperature was maintained at 35°C at both the right and

left sides. A gradient method with a mixture of water (A) and methanol (B) was used as follows: initial mix of 40% water, 60% methanol, linear increasing to 100% methanol within 5 min and held constant for 11 min, with a subsequent linear decrease to initial conditions (60% methanol) within 2 min, and finally a post-run at initial conditions for 10 min. The flow rate was 0.32 mL/min. An electrospray ionization (negative ion mode) mass spectrometer was used for the determination of triclosan and triclocarban.

3.8.4 Solids

TDS, TSS and VSS were measured according to the Standard Methods for the Examination of Water and Wastewater. For TDS, clean ceramic dishes were first placed in a 110°C oven (Thelco Laboratory oven) for at least one hour. After this time period, the dishes were removed, allowed to cool to room temperature and then weighed. After weighing, between 50 mL-250 mL of the sample was transferred to the ceramic dishes, and the dishes were placed back in the oven. After a few hours had elapsed (or the time required for the liquid samples to evaporate), the dishes were removed from the oven, allowed to cool and then weighed. The difference in weight as well as the sample volume were then used to compute the TDS.

For TSS and VSS measurement, clean aluminum dishes containing glass fiber filters were first placed in a 550°C oven (Thermo Scientific Lindberg blue m) for at least one hour. After this time period, the dishes and filters were removed and placed in the 110°C oven (Thelco Laboratory oven) for at least one hour. Following this, the dishes were removed and allowed to cool to room temperature and then weighed. After weighing, between 50 mL-250 mL of sample was filtered through the glass fiber filter. The filters were placed in the aluminum dishes and were placed back in the 110°C oven.

After a few hours had elapsed (or the required time for the filter to dry), the dishes were removed from the oven and then weighed. The difference in weight as well as the sample volume were then used to compute the TSS. The aluminum dishes and filters were then placed in the 550°C oven. After a few hours had elapsed (or the required time required for the solids to volatilize), the dishes were removed from the oven, allowed to cool to room temperature and then weighed. The difference in weight as well as the sample volume were then used to compute the VSS.

3.8.5 Nitrogen

3.8.5.1 Total Nitrogen *(referenced from Goldman and Clifford)*

The TN was measured using the same instrument used for TOC (TOC-VCSH Shimadzu organic carbon analyzer). For TN measurement, the Total Nitrogen Module (TNM-1) that conforms to the American Society of Test Method's (ASTM) D5176 is used. Chemiluminescence detection and combustion oxidation are used. The catalyst used is platinum at a temperature of 720°C. Conversion of bound nitrogen to nitrogen monoxide with further oxidation to nitrite occurs. The lower detection limit of the instrument is 0.05 ppm and the upper detection limit is 4000 ppm when used with an auto dilution feature.

3.8.5.2 Nitrate and Ammonium *(referenced from OI Analytical Flow Solution 3000 user manual)*

Nitrate and ammonium were measured using O.I Analytical Flow Solution 3000 (FS 3000; Figure 3.17). This is an automated high throughput system for ion analysis. Flow analysis techniques are applied for the ion analysis. The concentrations are measured using flow injection analysis. Flow Injection Analysis (FIA) techniques are typically used for the FS 3000. FIA is a recognized procedure in the Standard Methods

for the Examination of Water and Wastewater handbook. Samples are aspirated, reagents are added and a colorimetric or electrochemical reaction occurs and concentrations are measured in a continuously flowing stream.



Figure 3.17 O.I Analytical Flow Solution 3000

3.8.6 Salts, Boron and Sulfate (*referenced from Standard Methods, 2005 (3210 B)*)

The SAR serves as an index of the ratio of sodium (Na^+) to calcium (Ca^{2+}) and magnesium (Mg^{2+}) and is calculated as $[\text{Na}^+ / ((\text{Ca}^{2+} + \text{Mg}^{2+}) / 2)^{0.5}]$. It should be noted that the SAR was not calculated using the concentration in mg/L of the sodium, calcium and magnesium. Instead, the SAR was calculated using sodium, calcium, and magnesium concentration values normalized based on the molecular weight (MW) and valence charge of the ion (that is, using meq/L). For instance, Na^+ was calculated as

$$\frac{\text{concentration}(\text{mg} / \text{L})}{\left(\frac{\text{MW} = 23}{1} \right)} \text{ while } \text{Ca}^{2+} \text{ was calculated as } \frac{\text{concentration}(\text{mg} / \text{L})}{\left(\frac{\text{MW} = 40}{2} \right)}$$

Therefore, in order to determine the SAR, the calcium, magnesium and sodium concentrations had to be determined. These concentrations were measured using a TJA Solutions IRIS Advantage High Resolution Radial View Inductively Coupled Plasma

Atomic Emission Spectrometer (ICP-AES) following acid digestion of the leachate samples with nitric acid and hydrochloric acid. Boron and total sulfur were also measured by ICP-AES analysis following acid digestion of the leachate samples with nitric acid and hydrochloric acid. A flowing stream of argon gas ionized by an applied radio frequency field (oscillating at 27.1 MHz) makes up an ICP source. Excitation of atomic emission occurs as a result of the high temperature of the plasma. Ionic emission spectra is produced when a high percentage of the atoms are ionized. Low detection limits are possible with the ICP given the efficient excitation of atomic emission that occurs with the ICP.

3.8.7 Conductivity

The conductivity was simply measured using a ThermoOrion 145A+ Conductivity meter.

Chapter 4

RESULTS

4.1 Introduction

Setup of the greenhouse experiments was complete in October 2009. However, irrigation with synthetic graywater did not commence until February 2010. Prior to the start of graywater irrigation (between October 2009 and February 2010), all the plants and grasses were irrigated with potable water. Leachate sampling and analysis was performed once every two months from the onset of synthetic graywater irrigation. As of September 2010, there have been three sampling events occurring in April, June and August. For the sampling events, leachate was collected immediately after an irrigation event and analyzed for total organic carbon (TOC), total nitrogen (TN), nitrate, ammonium, boron, sodium adsorption ratio (SAR), total sulfur, conductivity, linear alkyl benzene sulfonate (LAS), alkyl ethoxy sulfate (AES), alkyl sulfate (AS), total dissolved solids (TDS), total suspended solids (TSS), and volatile suspended solids (VSS). Another analysis to be conducted is the measurement of alcohol ethoxylate (AE) (Chapter 1). However, to date, analysis of alcohol ethoxylate (AE) has not been completed. Therefore, these results are not provided in this chapter.

Although each plant and grass type had a total of eight pots (four for graywater irrigated and four for potable water irrigated; Chapter 3 – Section 3.3.1), samples were only collected from three pots each for each irrigation type (a total of six pots for

each plant or grass type). The results presented in this chapter are an average for three pots containing the same plant or grass type for each irrigation type. In the sampling event in June, leachate generation problems were observed for the graywater irrigated lemons, and one of the euonymous pots, such that analysis results in these cases are not reported. Leachate analysis for each irrigated plant and grass pot is provided in Appendix A, while average results are summarized in the subsequent sections of this chapter. It should be noted that the plant pots were sequentially numbered 1 through 8, with the first letter of either the plant or grass type preceding the number. For example, the first tall fescue pot is denoted as T1.

For each leachate analysis parameter, student's t-test was conducted to determine where differences were significant. Significance was done at 95% probability ($\alpha = 0.05$). Three sets of comparisons were made.

The first set involved a comparison at each sampling event (i.e. April, June, and August) between the leachate from the potable water irrigated and the graywater irrigated pots. The null hypothesis evaluated was that concentrations of each constituent were the same in leachate collected from graywater irrigated plant pots as potable irrigated plant pots. The three comparisons made for each analyte were:

- ❖ Graywater versus Potable water irrigation for April
- ❖ Graywater versus Potable water irrigation for June
- ❖ Graywater versus Potable water irrigation for August

The second and third null hypothesis evaluated were that there would be no difference in leachate constituent concentration for the various sampling dates (April, June and

August) for each graywater and potable water irrigated plant pot. The six comparisons for each analyte were:

- ❖ April versus June for Potable water and Graywater irrigation separately
- ❖ June versus August for Potable water and Graywater irrigation separately
- ❖ April versus August for Potable water and Graywater irrigation separately

4.2 Leachate Generation

Leachate generation volume was monitored through the experiment duration. Comparisons between the leachate generated from the potable water and graywater irrigated pots in terms of volume generated in addition to the physical and chemical characteristics of the leachate were made. Between October 2009 and February 2010, irrigation for both sets of pots (pots to be irrigated with graywater and the control pots) was done with potable water (Chapter 3 – Section 3.3.1). It was observed that from October 2009 to January 2010, the average leachate volume generated from all the pots were similar (Figures 4.1 to 4.4). The leachate generated from each individual pot is also provided in Appendix A. In February 2010, irrigation with potable water occurred in the first two weeks of the month for all pots. However, beginning in the third week of this month, irrigation with synthetic graywater commenced, with irrigation with the graywater only carried out on pots designated for use with graywater. It was observed that in February 2010, the leachate volume for the graywater and the potable water irrigated pots were similar. However, in March 2010, the leachate volume generated from the graywater irrigated pots was less than that generated from the potable water irrigated pots.

The trend of lower leachate volume for the graywater irrigated pots compared to the potable water irrigated pots was observed for both the plants and grasses. As of September 2010, this trend has still been observed. Statistical analysis revealed that in the months of May, June, July, and August, there was a significant difference ($p < 0.05$) between the leachate volume generated from the graywater irrigated pots and the potable water irrigated pots for lemon, tall fescue and bermudagrass. For bermudagrass, there was also significant difference ($p < 0.05$) between the graywater irrigated pots and the potable water irrigated pots in March and April. In June, July and August, there was significant difference ($p < 0.05$) between the graywater irrigated pots and the potable water irrigated pots for euonymous.

Several factors can be attributed to the reduction in leachate volume for the graywater irrigated pots. At the forefront is the uptake of a larger volume of water by the graywater irrigated plants and grasses. The graywater irrigated plants have larger leaf size and canopy cover, and the grasses have a larger canopy cover as well (Section 4.15) thus leading to larger uptake of water. With larger water uptake by plants and grasses, the leachate volume will subsequently be decreased. Another factor to be considered is the change in the soil matrix and infiltration capacity. When the infiltration ability of the soil is compromised, more time will be required for the water to percolate downgradient through the soil column, and thus the leachate volume will be reduced.

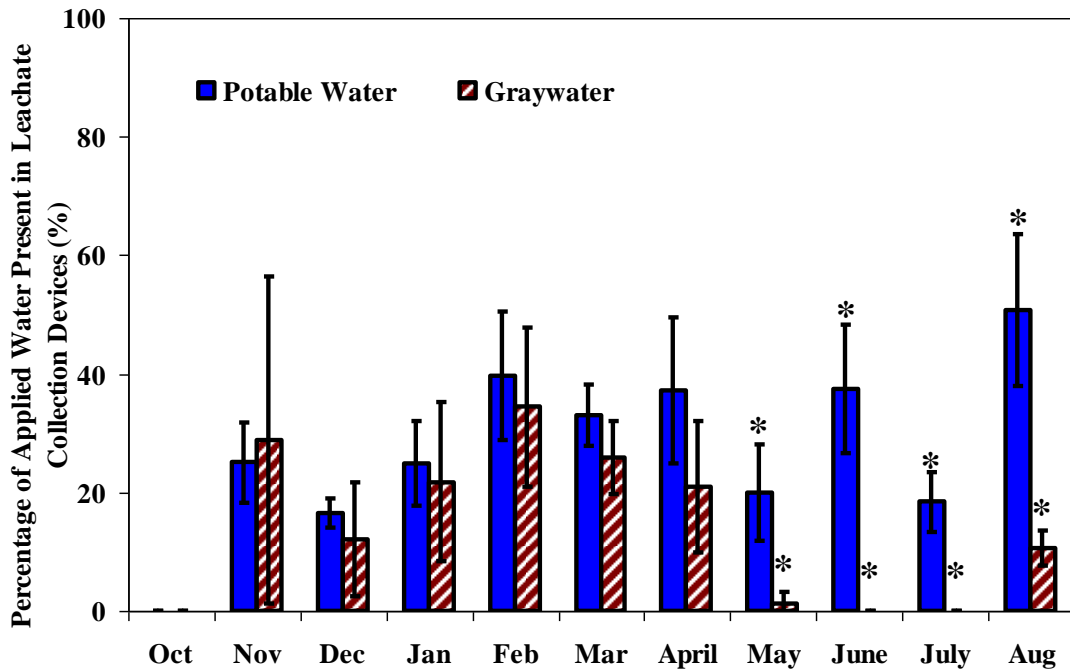


Figure 4.1 Percentage of Leachate Generated for Lemon
 (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated in one month

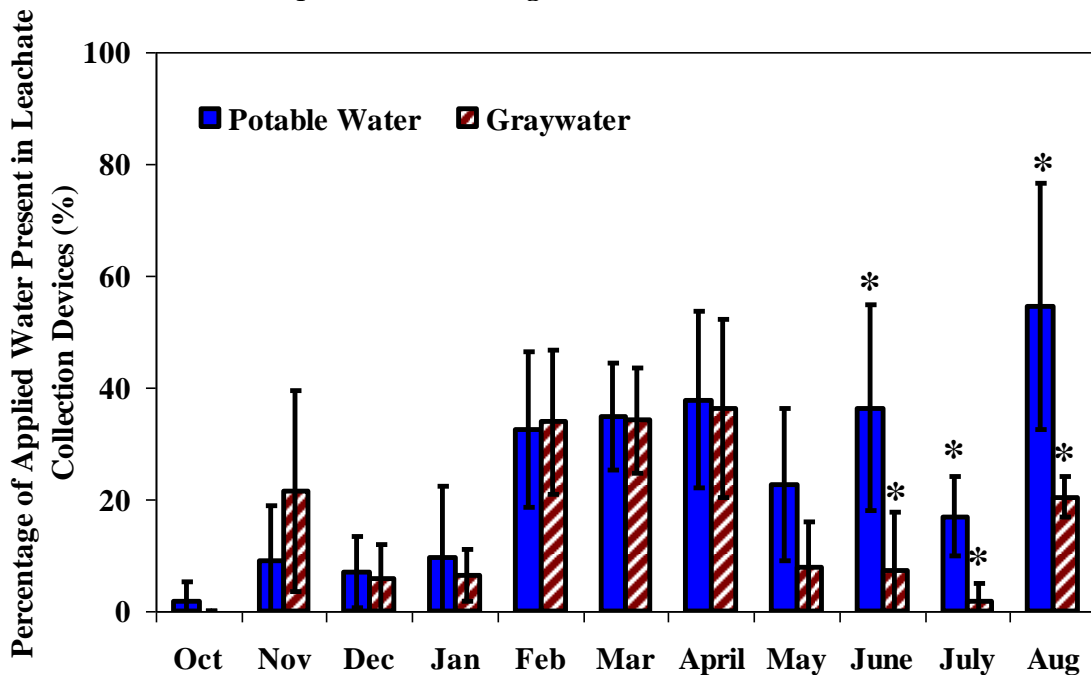


Figure 4.2 Percentage of Leachate Generated for Euonymous
 (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated in one month

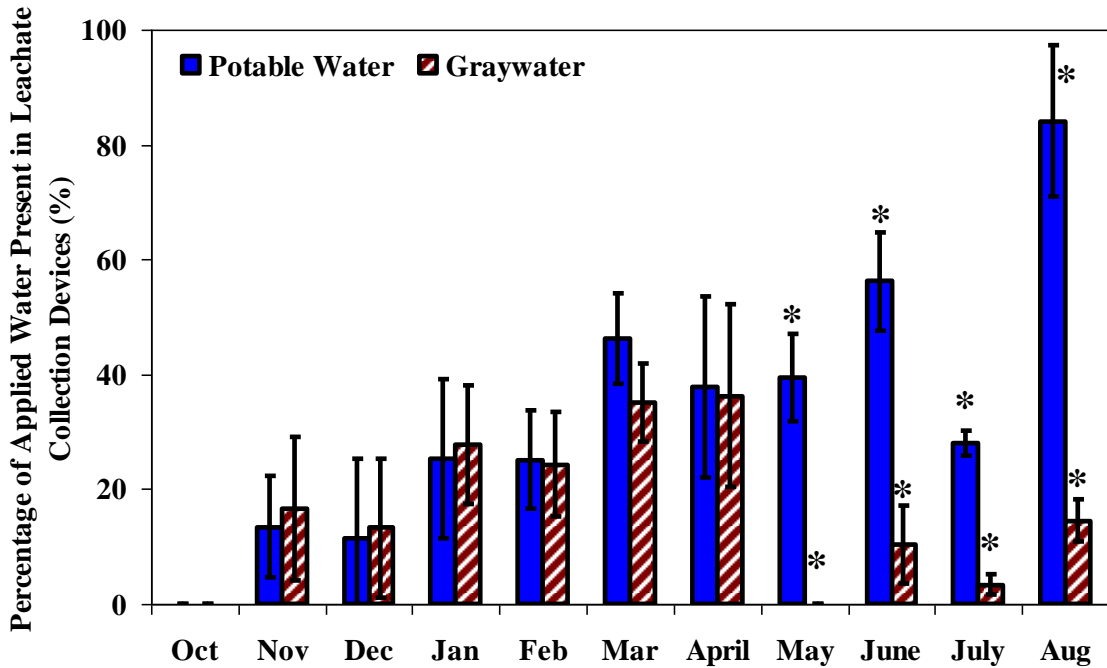


Figure 4.3 Percentage of Leachate Generated for Tall Fescue
 (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated in one month

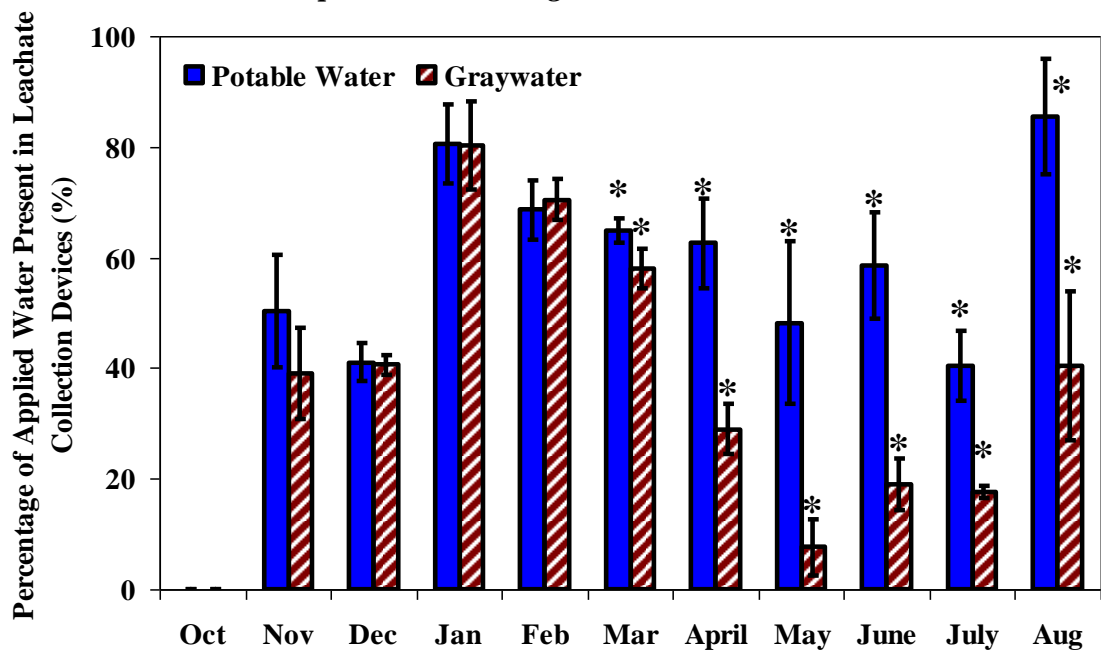


Figure 4.4 Percentage of Leachate Generated for Bermudagrass
 (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated in one month

A noteworthy consideration that has presently not been evaluated is the alteration of the soil matrix and changes to the soil stability. These two factors can be attributed to the presence of elevated levels of surfactants and sodium (Abu-Zreig et al. 2003; Ayres and Westcott, 1985). Changes in the sodium concentration and subsequent SAR can have adverse effects on the soil stability. The surface soil can be weakened by high sodium, which weakens the soil structure, results in surface soil aggregation and dispersion to smaller particles resulting in clogging of soil pores (Ayres and Westcott, 1985).

4.3 Total Organic Carbon

Leachate analysis for the TOC revealed that in the months of April, June and August, the TOC measured in the leachate from the graywater irrigated pots was higher than that measured in the leachate from the potable water irrigated pots for tall fescue and bermudagrass (Figure 4.5). In April and August, for lemon, the TOC was higher in the leachate from the graywater irrigated pots compared to the leachate from the potable water irrigated pots (Figure 4.6). The exception to the trend of higher measured TOC in the leachate from the graywater irrigated pots occurred for euonymus where the TOC in the leachate from the graywater irrigated pots was lower compared to the TOC in the leachate from the potable water irrigated pots in April and June, but the reverse was the case in August.

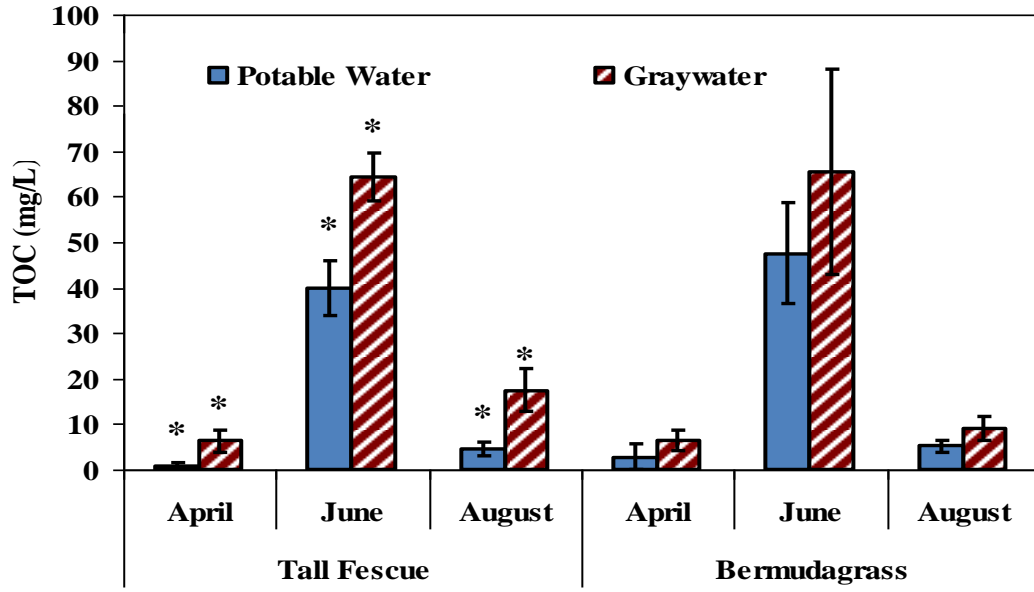


Figure 4.5 TOC for Tall Fescue and Bermudagrass (synthetic graywater TOC: 84.91 mg/L) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

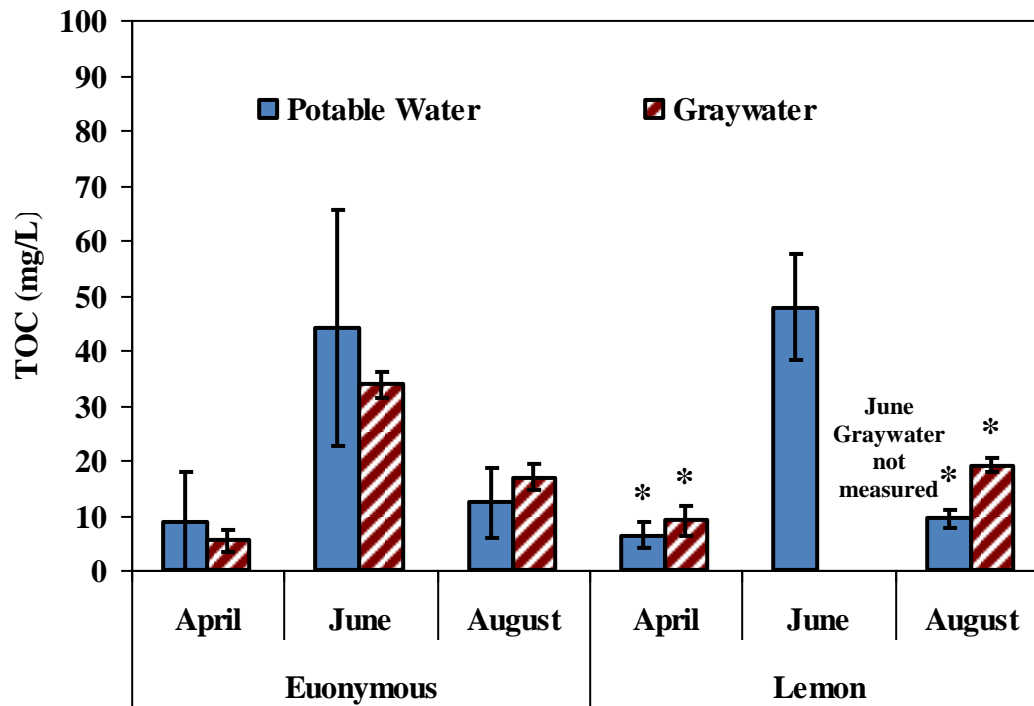


Figure 4.6 TOC for Euonymous and Lemon (synthetic graywater TOC: 84.91 mg/L) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

Statistical analysis revealed that despite the higher measured concentrations of TOC in the leachate from the graywater irrigated pots, the difference between the TOC in the leachate from the graywater irrigated and the potable water irrigated pots was only significant ($p < 0.05$) in the case of tall fescue and lemon. Compared to April and August, the TOC measured in June was significantly higher ($p < 0.05$) for the plants and grasses. In June, the total carbon was similar to that measured in April and August, but the inorganic carbon was lower thus accounting for increased TOC. In just considering the TOC between April and June for the graywater irrigated plant pots, it was observed that for all the plants and grasses there was an increase in the TOC between these months (Figures 4.5 and 4.6). This increase was significant ($p < 0.05$) except in the case of tall fescue. No specific trends were noted for the TOC concentration in the leachate from the graywater irrigated pots over the various sampling events.

The TOC was also measured for the case of irrigation of only soil (no plant or grass; Figure 4.7). There was no significant difference ($p > 0.05$) noted between the TOC in the leachate from the graywater irrigated pots and the potable water irrigated pots in the months of June and August. It was further found that in August, the TOC in the leachate from the potable water irrigated and graywater irrigated pots were identical.

In comparing the results for the case of only soil (no plants) to the case where plants and grasses were present, in June, the soil only scenario had higher TOC in the leachate from the graywater irrigated pots compared to the TOC measured in the leachate from the graywater irrigated euonymous pots. Conversely, the TOC measured in the leachate from the graywater irrigated soil with no plants or grasses present was lower than the TOC measured in the leachate from the graywater irrigated tall fescue and

bermudagrass. However, in August, the TOC in the leachate from the graywater irrigated soil with no plants or grasses was lower compared to TOC measured in the leachate from the graywater irrigated lemon, euonymous, tall fescue and bermudagrass. These results indicate that the source of TOC is not only from graywater, but also likely from plant root exudates.

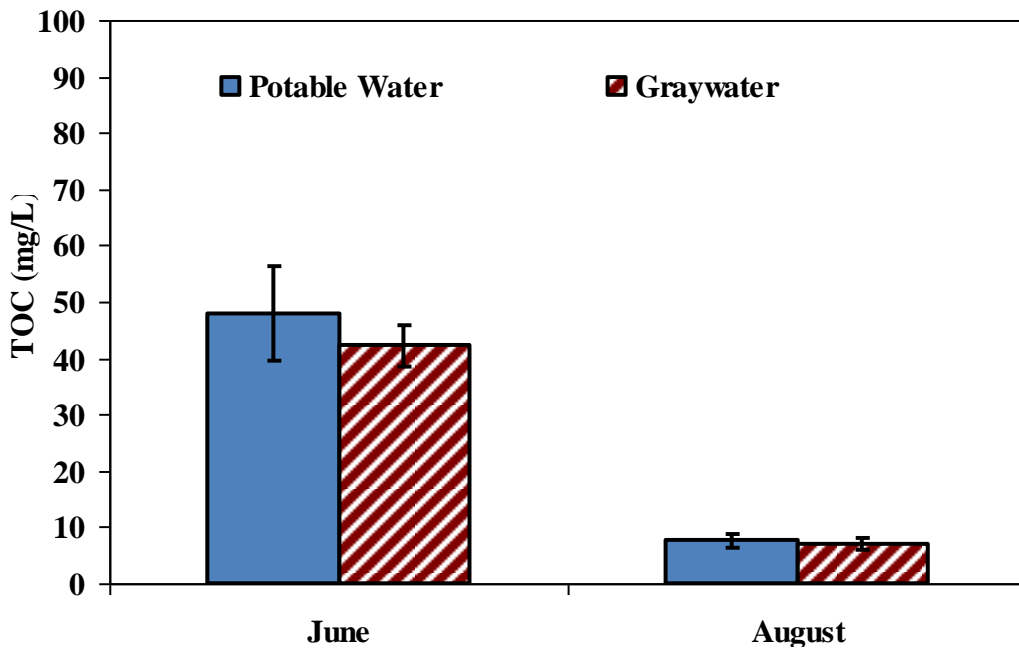


Figure 4.7 TOC in Leachate Collected from Pots with No Plants (*synthetic graywater TOC: 84.91 mg/L*)

It was determined that in April, for the graywater irrigated pots, on average, 92.4% of the TOC was retained in the soil column for bermudagrass, with the percentage retained of TOC for euonymous, lemon and tall fescue being respectively 93.8%, 89.4% and 92.6%. (Table 4.1). In June, for the graywater irrigated pots, the percentage retained for TOC was 22.9%, 60.4%, and 24.2% for bermudagrass, euonymous, and tall fescue respectively. It was further observed that in June, similar to April (except for lemon in

April), there was less TOC retention in the soil column for grasses than for the plants. In August, for the graywater irrigated pots, for bermudagrass, euonymous, lemon and tall fescue, the percentage TOC retained was on average 89.5%, 80.2%, 77.6% and 79.4% respectively. Contrary to the trend noted in April and June, in August, there was more TOC retention through the grass columns compared to the columns containing the plant biomass. In the case of the graywater irrigated pots with no plants or grasses present, in June and August, the percentage retained was on average 50.3% and 91.7% respectively, thus indicating that in these sampling months there was more retention of organics in the soil column when no plants were present compared to when plants and grasses were present (except for euonymous in June).

Table 4.1 Percentage Retained for Some Graywater Constituents

		Percentage Retained (%)			
		TOC	LAS	AS/AES	TN
Tall Fescue	April	92.56	98.75	97.31	77.2
	June	24.23	94.99	97.61	77.8
	August	79.43	¹ NM	¹ NM	78.2
<hr/>					
Bermudagrass	April	92.39	98.08	97.33	70.4
	June	22.88	96.78	96.65	59.7
	August	89.51	¹ NM	¹ NM	76.4
<hr/>					
Euonymous	April	93.76	98.15	97.40	41.1
	June	60.38	94.50	90.85	22.8
	August	80.23	¹ NM	¹ NM	46.8
<hr/>					
Lemon	April	89.43	95.65	97.46	36.4
	June	¹ NM	¹ NM	¹ NM	¹ NM
	August	77.63	¹ NM	¹ NM	47.8

¹NM = Not Measured

4.4 Surfactants

4.4.1 Linear Alkylbenzene Sulfonate

LAS measured in the leachate from the graywater irrigated pots was always lower than that present in the synthetic graywater (21.0 mg/L; Figures 4.8 and 4.9), indicating that there was removal of LAS in the pots irrigated with graywater through either biodegradation, plant uptake or sorption to soil.

In April and June, LAS measured in the leachate from the graywater irrigated pots was higher than that measured in the leachate from the potable water irrigated pots for euonymous, tall fescue, bermudagrass and lemon (Figures 4.8 and 4.9). It was also found that between April to June, there was an increase in the measured LAS concentration in the leachate from the graywater irrigated pots for tall fescue, bermudagrass and euonymous, while not statistically significant ($p > 0.05$).

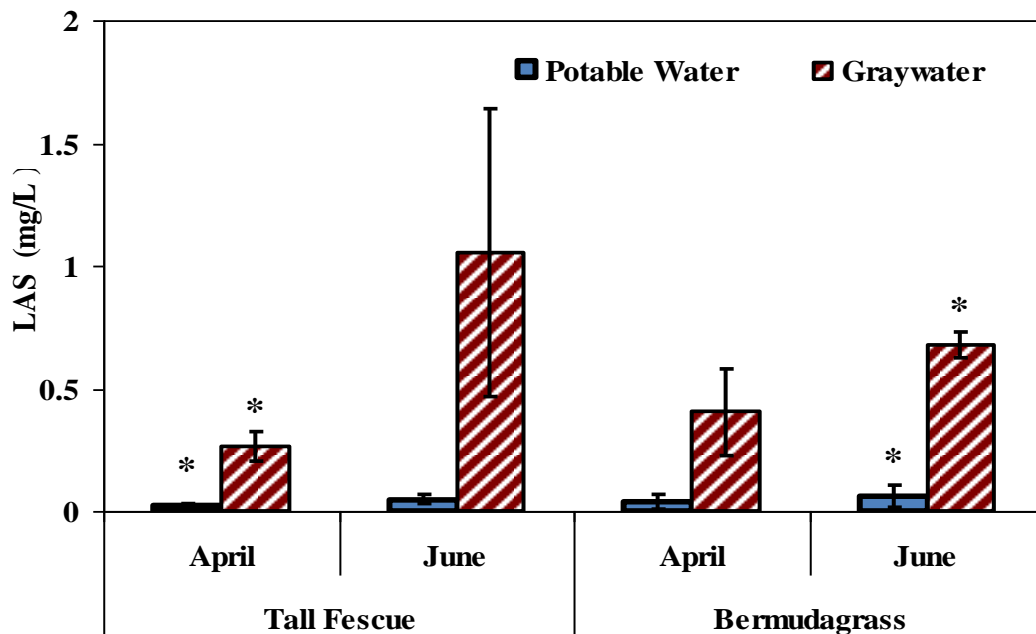


Figure 4.8 LAS for Tall Fescue and Bermudagrass (*synthetic graywater LAS: 21.0 mg/L*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April or June)

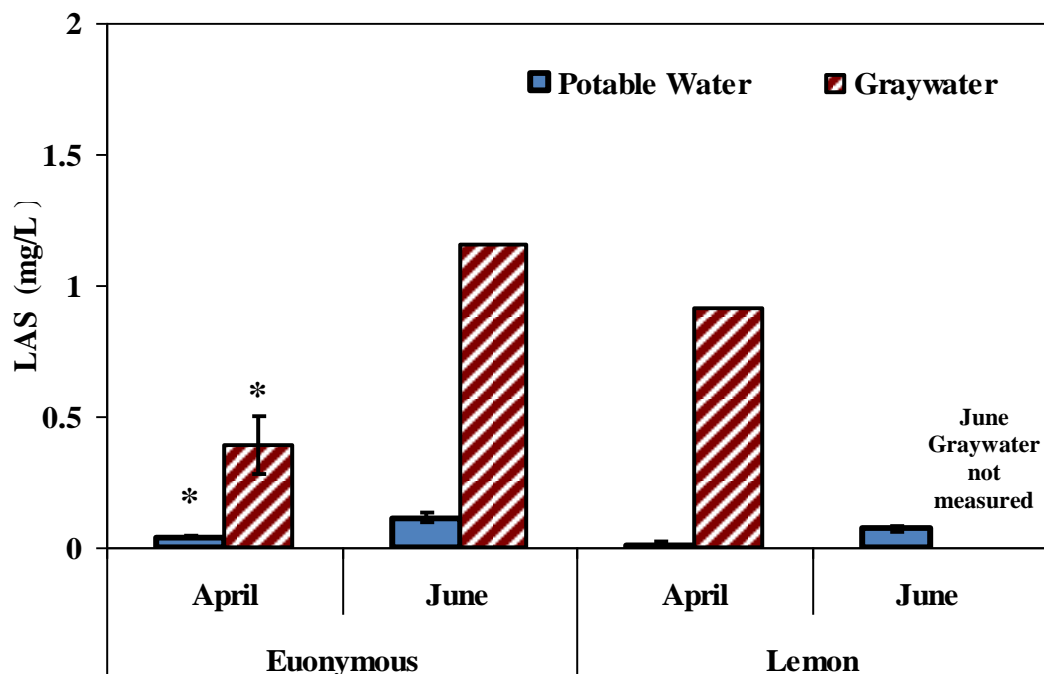


Figure 4.9 LAS for Euonymous and Lemon (synthetic graywater LAS: 21.0 mg/L; Replicate samples unavailable in June for Euonymous and April for Lemon) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April or June)

The LAS concentrations in April for tall fescue and euonymous were found to be significantly different ($p < 0.05$) when comparisons were made between leachate generated from pots irrigated with potable water and graywater. Likewise, statistically significant difference ($p < 0.05$) was also determined for bermudagrass in June.

Of note is that in June for euonymous and in April for lemon, the difference in the measured LAS in the leachate from the graywater irrigated pots and the potable water irrigated pots would most likely be statistically significant. However, for the euonymous and lemon in the months formerly stated, determination of significant difference could not be made. This is because in April, for the graywater irrigated lemon, it is believed that based on the LAS concentrations measured in two of the lemon pots compared to the third sampled pot, there was an error in the analysis. Therefore, enough data points were

unavailable to carry out statistical analysis. Likewise, for the leachate from the graywater irrigated euonymous pots, there was only one data point, hence statistical analysis could not be performed.

For the case of irrigation of soil columns containing no plants or grasses (soil only), the LAS in the leachate from the graywater irrigated pots was higher than that measured in leachate from the potable water irrigated pots (Figure 4.10). In both April and June, the LAS measured in the leachate from the graywater irrigated pots for the case of no plants or grasses was less than that measured in the leachate from the graywater irrigated pots when plants and grasses were present. As was the case for the leachate from the graywater irrigated plants and grasses, the LAS in the leachate from the graywater irrigated pots with only soil was higher in June compared to April. However, this difference was not statistically significant ($p > 0.05$).

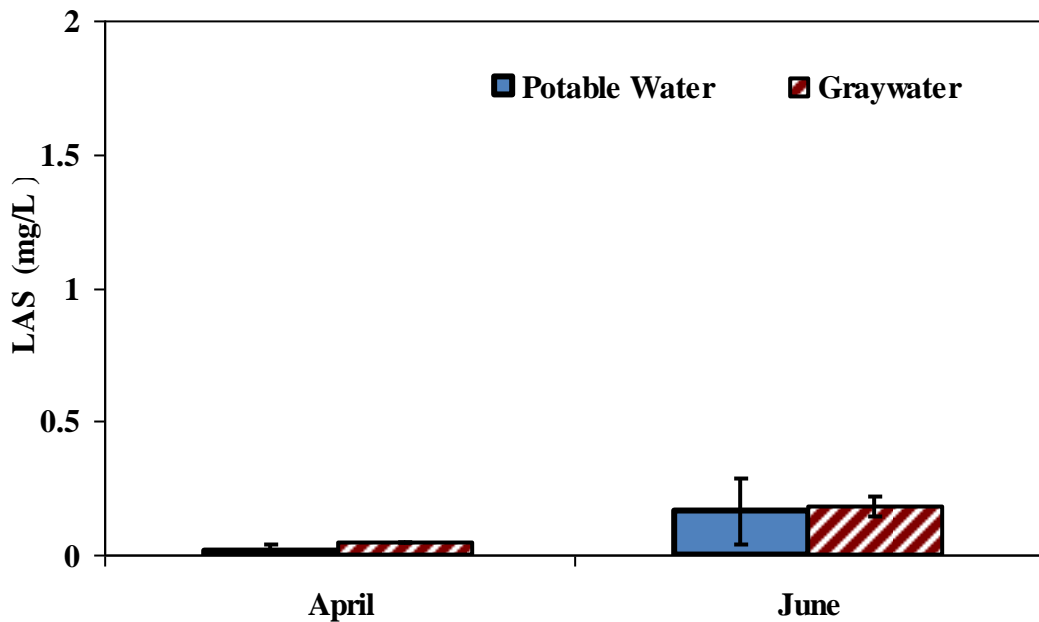


Figure 4.10 LAS in Leachate Collected from Pots with No Plants (*synthetic graywater LAS: 21.0 mg/L*)

Although high sorption coefficients have often been reported for LAS (Chapter 1 – Section 1.4.7.1), sorption of LAS to soil is also affected by other factors including the soil type and the presence of organic matter in the soil. It is usually expected that there is faster degradation of LAS for a plant system given the biotransformation that can occur in the rhizosphere (Chapter 1 – Section 1.4.7.4). However, given that higher LAS concentrations were measured in the leachate from the graywater irrigated plants and grasses compared to the leachate from the graywater irrigated soil with no plants and grasses, the implication is that degradation by microbial action in the rhizosphere does not occur. It should however be noted that given the LAS concentration of 21 mg/L, phytotoxicity as a result of LAS will not occur. This is because published literature has revealed that substantially higher surfactant concentrations would have to be present in the plant system before phytotoxic effects are observed (concentrations greater than 250 mg/L; Bubenheim et al. 1997).

For the pots with plants and grasses, the plant roots result in the penetration of LAS. The residence time for LAS may be shortened in these plant systems leading to less time for biodegradation. If biodegradation does not occur coupled with migration pathways created in the soil by plant roots, LAS will be leached out. Reduction in biodegradation ability might be attributed to fewer microbes present in the root zone for the greenhouse plants than would typically be seen under normal field conditions.

It was determined that of the 21 mg/L of LAS applied to the graywater irrigated pots, only 1.92% of the LAS was measured in the leachate from the graywater irrigated bermudagrass in April, thus implying that the percentage retained of the LAS was 98.08%. For a predominance of sorption, the implication is that on average,

approximately 98% (simply denoted as the retention percentage) of the LAS is retained in the soil column for the case of the graywater irrigated bermudagrass. Of the original 21 mg/L LAS in the graywater, for the leachate from the graywater irrigated tall fescue, euonymous and lemon, 1.25%, 1.85% and 4.35% of the original LAS concentration was respectively measured in the leachate thus implying sorption, biodegradation or plant uptake of 98.75%, 98.15% and 95.65% through the soil column for the tall fescue, euonymous and lemon respectively. In June, retention percentages of LAS for the graywater irrigated plants and grasses decreased with measured values of 96.78%, 94.50%, and 94.99% for bermudagrass, euonymous, and tall fescue respectively (Table 4.1) thus implying an increase in the leaching potential of the LAS.

4.4.2 Alkyl Ethoxy Sulfate and Alkyl Sulfate

Consistent with LAS results, AES and AS in the leachate from the graywater irrigated pots with plants and grasses was always lower than the AS/AES present in the synthetic graywater (4.5mg/L; Figures 4.11 and 4.12), indicating that there was either removal of AS/AES in plant pots irrigated with graywater through biodegradation, retention by plant roots or sorption to soil.

In April and June, AES and AS measured in the leachate generated from graywater irrigated pots were higher than that measured in the leachate generated from potable water irrigated pots for euonymous, tall fescue, bermudagrass and lemon (Figures 4.11 and 4.12). In April and June, for tall fescue and bermudagrass, comparisons between the AS and AES in the leachate from the graywater irrigated and potable water irrigated

pots revealed that there was significant difference ($p < 0.05$) between these two measured concentrations.

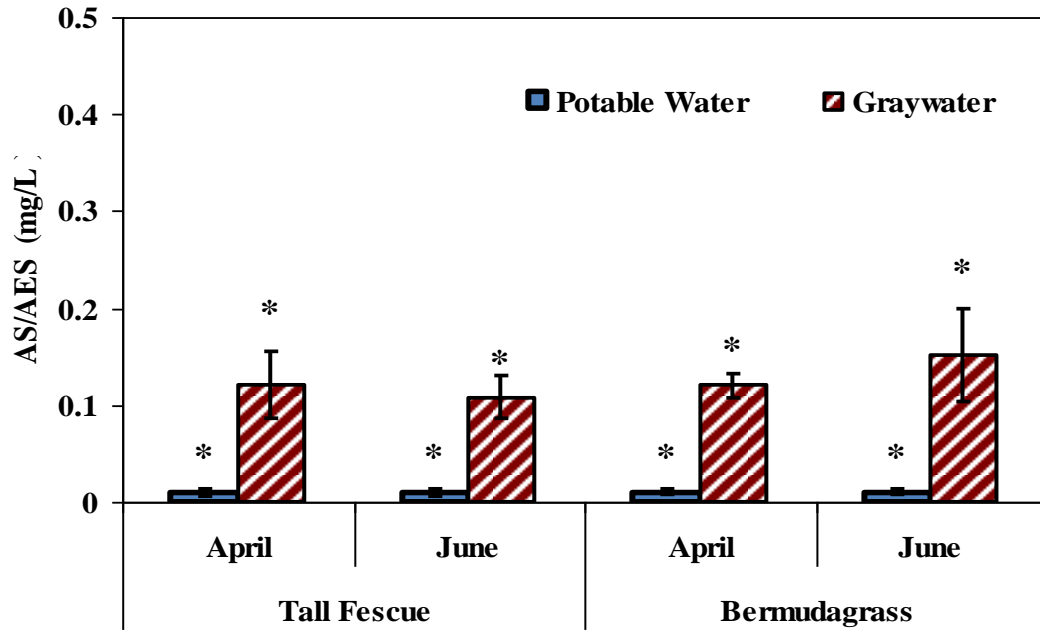


Figure 4.11 AS/AES for Tall Fescue and Bermudagrass (synthetic graywater AS/AES: 4.5 mg/L) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April or June)

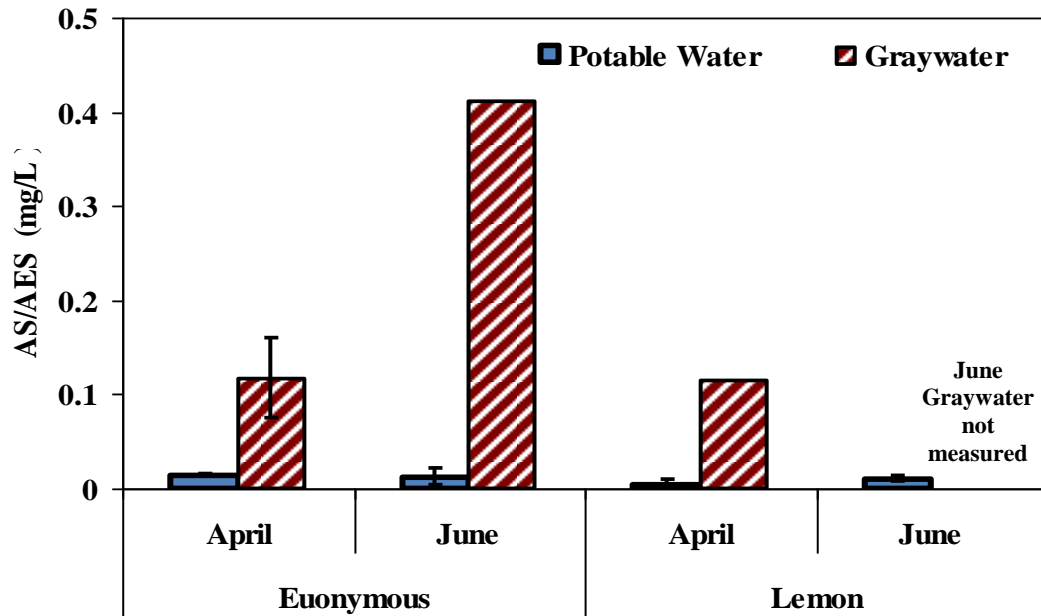


Figure 4.12 AS/AES for Euonymous and Lemon (synthetic graywater AS/AES: 4.5 mg/L; Replicate samples unavailable in June for Euonymous and April for Lemon)

Of note is that in June for euonymous, the difference in the measured AS/AES in the leachate from the graywater irrigated pots and the potable water irrigated pots would most likely be statistically significant. However, for the euonymous in June, determination of significant difference could not be made. For the leachate from the graywater irrigated euonymous pots, there was only one data point; hence, statistical analysis could not be performed. In April for lemon, statistical analysis could not be performed. This is because in April, for the graywater irrigated lemon, it is believed that based on the AS/AES concentrations measured in two of the lemon pots compared to the third sampled pot, there was an error in the analysis. Therefore, enough data points were unavailable to carry out statistical analysis.

While AS/AES in the leachate generated from the graywater irrigated euonymous and bermudagrass pots did increase from April to June (Figures 4.11 and 4.12), other data did not indicate an increase in AS/AES concentration with duration of graywater irrigation, with no significant difference found ($p > 0.05$). In both April and June, the AS/AES measured in the leachate generated from the graywater irrigated plants and grasses was higher than that measured in the leachate from the graywater irrigated pots with no plants or grasses (Figure 4.13).

AES has a lower sorption coefficient compared to LAS (Chapter 1 – Section 1.4.7.1). This would imply that the leaching potential of AES compared to LAS would be higher. Consistently, the measured AS/AES in the leachate from the graywater irrigated pots was less than the LAS measured in the leachate from the graywater irrigated pots. This trend of lower measured AS/AES concentrations in comparison to LAS can however be attributed to the lower concentration of AS/AES in the graywater. The mechanism

hypothesized for LAS could also be adapted to the behavior of AS/AES. Given that higher concentrations of AS/AES were measured in the leachate from the graywater irrigated plants and grasses than for the graywater irrigated soil only (with no plants or grasses), the implication is that degradation of AS/AES by microbial action does not occur. Biodegradation as the predominant pathway for AS/AES should result in similar concentrations for the AS/AES measured regardless of the presence or absence of plants. Plant roots therefore affect the AS/AES that is retained in the soil system.

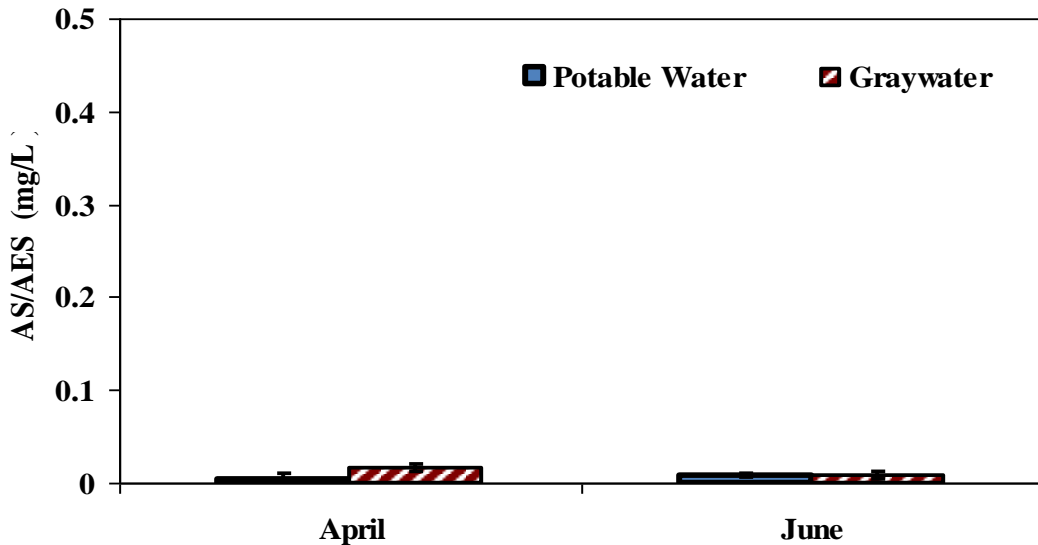


Figure 4.13 AS/AES in Leachate Collected from Pots with No Plants (*synthetic graywater AS/AES: 4.5 mg/L*)

There was higher percentage recovery of AS/AES in the leachate from the graywater irrigated pots (except for the case of lemon in April and tall fescue in June) when compared to the percentage recovery of LAS in the leachate from the graywater irrigated pots, thus suggesting the higher leaching potential of AS/AES compared to LAS. It was determined that of the 4.5 mg/L of AS/AES applied to the graywater irrigated pots,

2.67% of the AS/AES was measured in the leachate from the graywater irrigated bermudagrass in April, thus implying that 97.33% of the AS/AES was either retained in the soil column through sorption mechanisms or might have biodegraded or been taken up by plants (Table 4.1). For a predominance of sorption, the implication is that on average, approximately 97% of the AS/AES was retained in the soil column for the case of the graywater irrigated bermudagrass. Of the original 4.5 mg/L AS/AES in the graywater, for the leachate from the graywater irrigated tall fescue, euonymous and lemon, 2.69%, 2.60% and 2.54% of the original AS/AES concentration was respectively measured in the leachate thus implying retention percentages of 97.31%, 97.40% and 97.46% through the soil column for the tall fescue, euonymous and lemon respectively. In June, the percentage retained of AS/AES for the graywater irrigated plants and grasses were 96.65%, 90.85%, and 97.61% for bermudagrass, euonymous, and tall fescue respectively.

4.5 Total Sulfur (Sulfate)

The total sulfur (TS) was measured in the collected leachate samples. Given that the samples were filtered prior to sulfur measurement, and contributions of sulfur in the graywater irrigated systems comes from sulfate, the measured sulfur can be considered as predominant contributions of sulfate. The TS in the leachate collected from the graywater irrigated pots was found to be higher than that measured in the leachate from the potable water irrigated pots in the sampling months for both the plants and grasses (Figures 4.14 and 4.15).

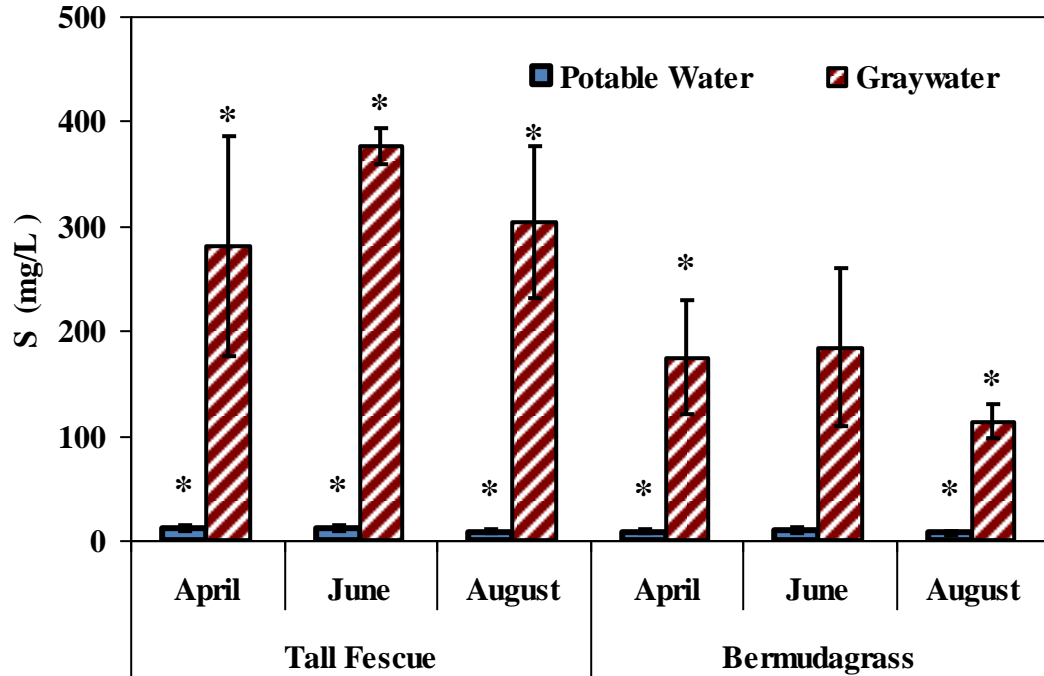


Figure 4.14 Total Sulfur for Tall Fescue and Bermudagrass (*synthetic graywater Sulfate: 174.32 mg/L = 58.11 mg/L as S*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

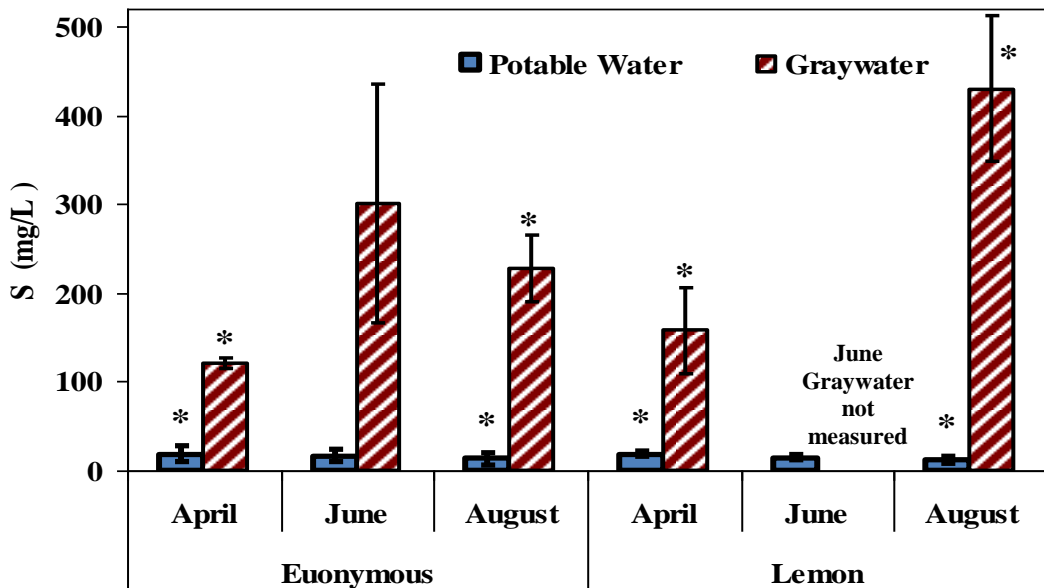


Figure 4.15 Total Sulfur for Euonymous and Lemon (*synthetic graywater Sulfate: 174.32 mg/L = 58.11 mg/L as S*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

In April, June and August, the difference between the TS in the leachate from the graywater and potable water irrigated pots for tall fescue was found to be significant ($p < 0.05$). In April and August, significant difference ($p < 0.05$) for the TS in the leachate from the graywater and the potable water irrigated pots was also determined for bermudagrass, lemon and euonymous. It was observed that compared to the graywater irrigated pots containing plants and grasses, the TS measured in the leachate from the graywater irrigated pots for the case of no plants or grasses was less than that measured when there were plants and grasses present (Figure 4.16). This trend corresponds to the trend noted for both LAS and AS/AES (sulfate based surfactants) where the surfactant concentration measured in the leachate from the graywater irrigated pots was less when there were no plants or grasses present. The TS measured in the leachate from the graywater irrigated pots when no plants or grasses were present decreased from 104 mg/L in April to 59 mg/L in August. Despite the high concentration of 104 mg/L measured in April for the case where no plants or grasses were present, the range of concentrations over the three sampling months was still less compared to the TS measured in the leachate from the graywater irrigated pots when plants and grasses were present, where concentrations as high as 429 mg/L were measured.

In all the sampling months and for the different scenarios evaluated for the graywater irrigated pots, the measured TS in the leachate was always higher than the TS in the graywater. Given the substantially higher TS measured in the leachate from the graywater irrigated pots when plants and grasses were present, it is hypothesized that the elevated TS levels can be attributed to the applied fertilizer (Chapter 3 – Section 3.6) in pots containing plants and grasses coupled with accumulation of sulfate in the soil.

However, given the substantially higher TS levels in the leachate from the graywater irrigated pots where plants and grasses were present compared to the graywater irrigated pots with no plants or grasses, it is believed that there is a mechanism of gradual release of the TS primarily from the fertilizer pellets through the soil profile though no discernable trends in the measured TS with time could be ascertained.

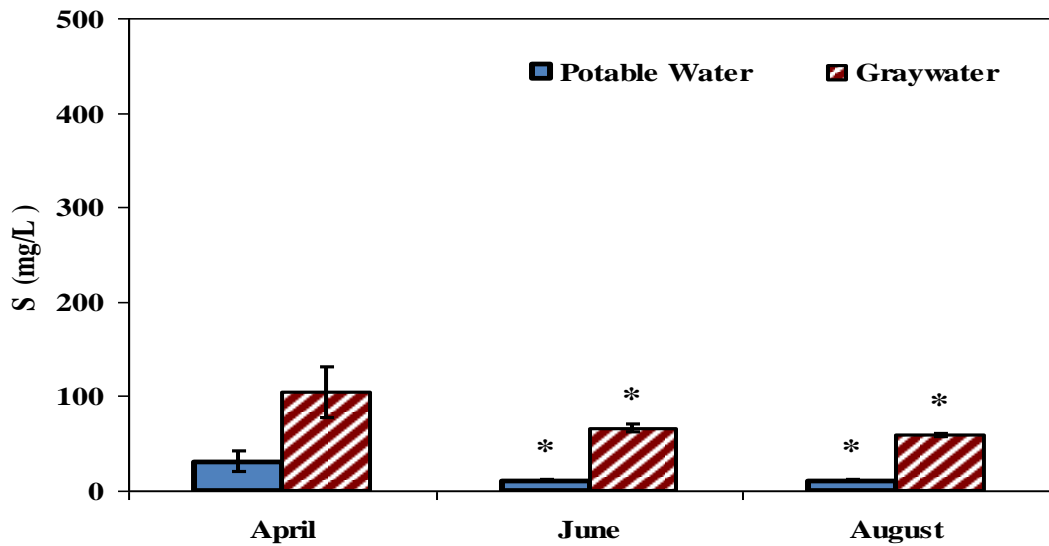


Figure 4.16 Total S in Leachate Collected from Pots with No Plants (*synthetic graywater Sulfate: 174.32 mg/L = 58.11 mg/L as S*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

4.6 Boron

The boron concentration was consistently higher in the leachate from pots irrigated with graywater compared to pots irrigated with potable water. This difference was significant ($p < 0.05$) in June and August for bermudagrass and euonymous but only in August for lemon (Figures 4.17 and 4.18).

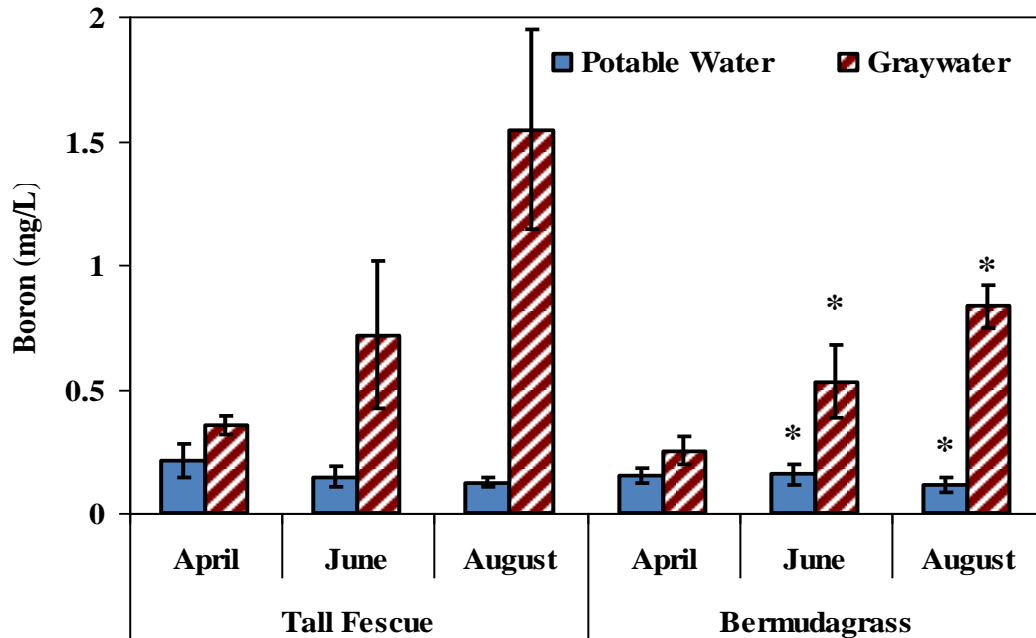


Figure 4.17 Boron for Tall Fescue and Bermudagrass (*synthetic graywater boron: 0.50 mg/L*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

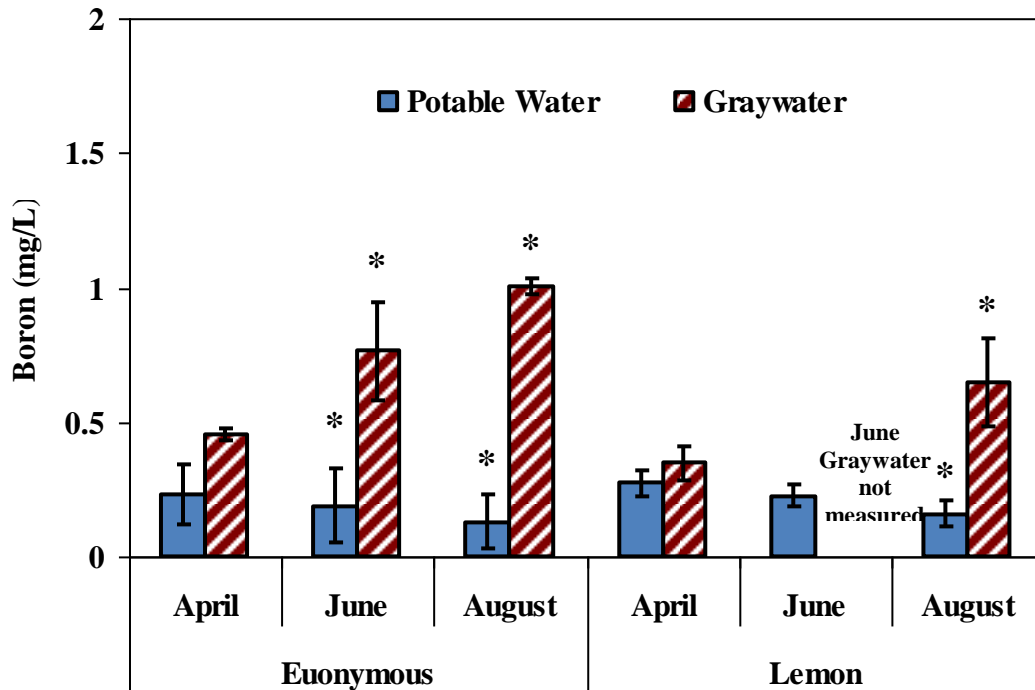


Figure 4.18 Boron for Euonymous and Lemon (*synthetic graywater boron: 0.50 mg/L*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

Boron was consistently present in the leachate from pots irrigated with potable water, likely a result of the presence of boron in the soil used for this study (55.4 ppm; Chapter 3 – Section 3.3.2). For the graywater irrigated soil with no plants or grasses, it was found that in April, the measured boron concentration was approximately the same as that measured for the potable water irrigated pots (0.37 mg/L and 0.35 mg/L for potable water and graywater respectively; Figure 4.19).

In June and August, significant difference ($p < 0.05$) was determined for the leachate from the graywater irrigated pots with only soil compared to the leachate from the potable water irrigated pots with soil. In both April and June, the measured boron concentration for the leachate from only the soil irrigated with graywater (no plants or grasses) was less than the measured concentrations in the leachate from the graywater irrigated tall fescue (0.35 mg/L and 0.72 mg/L) and euonymous (0.45 mg/L and 0.76 mg/L) pots. Conversely, the measured boron concentration for the leachate from the graywater irrigated pots with only soil was higher than the boron measured for the leachate from the bermudagrass (0.25 mg/L and 0.53 mg/L) and lemon (0.35 mg/L) pots. In August, the boron concentration measured in the leachate from the pots with no plants irrigated with graywater (0.46 mg/L) was less than the boron measured in the leachate from the graywater irrigated tall fescue (1.54 mg/L), bermudagrass (0.83 mg/L), euonymous (1.00 mg/L) and lemon (0.65 mg/L) pots.

It was further observed that there was a progressive increase in the boron measured in the leachate from the graywater irrigated euonymous, bermudagrass, lemon and tall fescue such that by June, accumulation above the boron concentration in the synthetic graywater (0.50 mg/L) had occurred. It was further found that between April to

August, the increase in boron concentration for bermudagrass and euonymous was significant ($p < 0.05$). Boron accumulation has previously been reported (Shafran et al. 2006, Gross et al. 2005). The increase in the measured boron concentration in the leachate indicates that there is accumulation of boron occurring and subsequent progressive leaching of the boron with time.

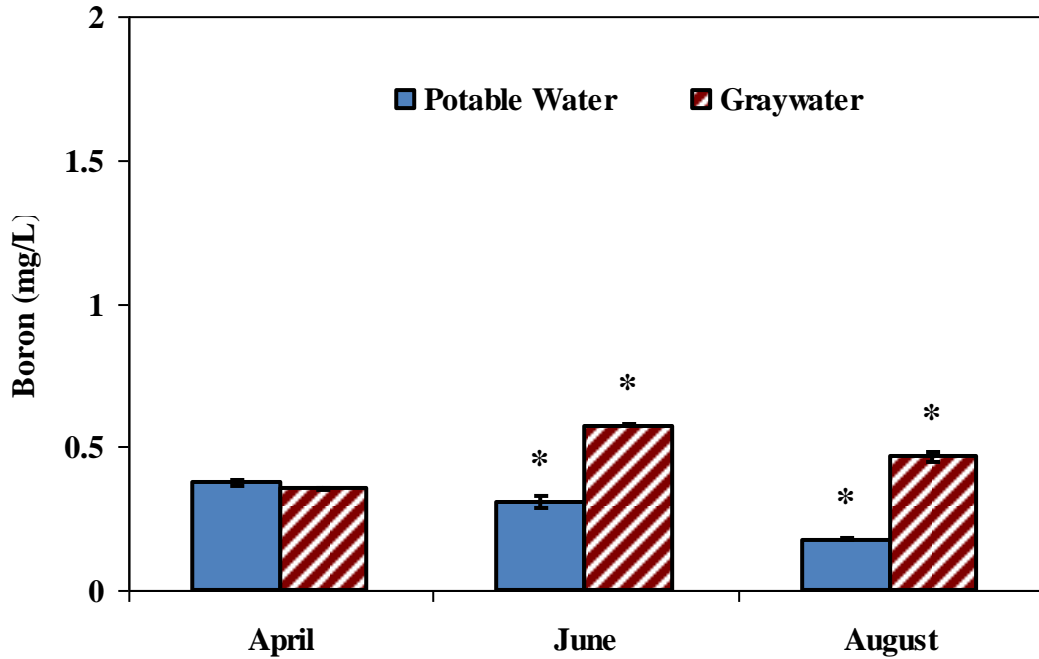


Figure 4.19 Boron in Leachate Collected from Pots with No Plants (*synthetic graywater boron: 0.50 mg/L*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

4.7 Total Nitrogen

Analysis of the TN in the collected leachate revealed that in the months of April, June and August, the TN measured in the leachate from the graywater irrigated pots was

higher than the TN measured in the leachate from the potable water irrigated pots for tall fescue, bermudagrass, euonymous and lemon (Figures 4.20 and 4.21).

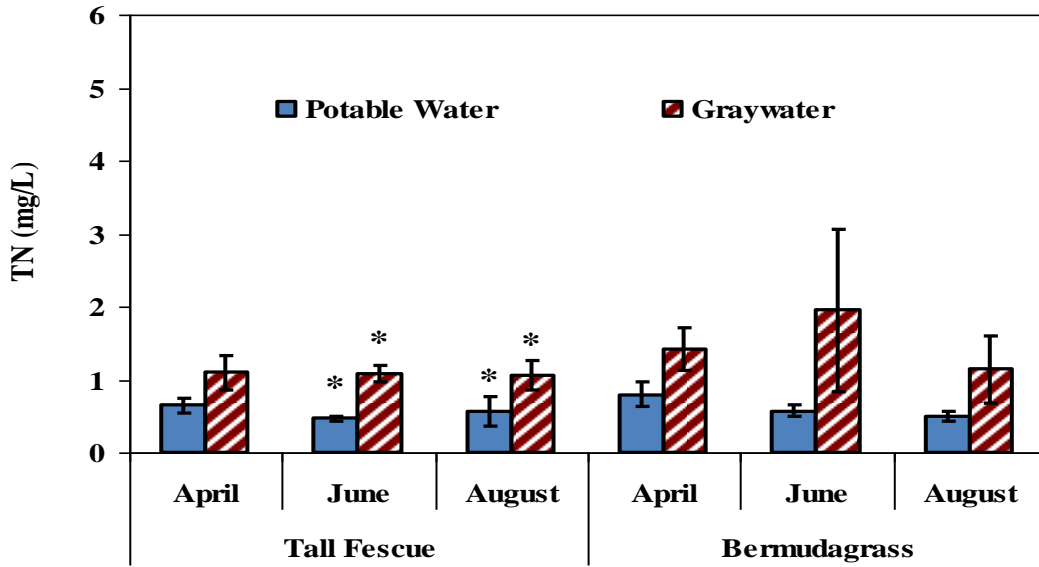


Figure 4.20 TN for Tall Fescue and Bermudagrass (*synthetic graywater TN: 4.82 mg/L as N*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

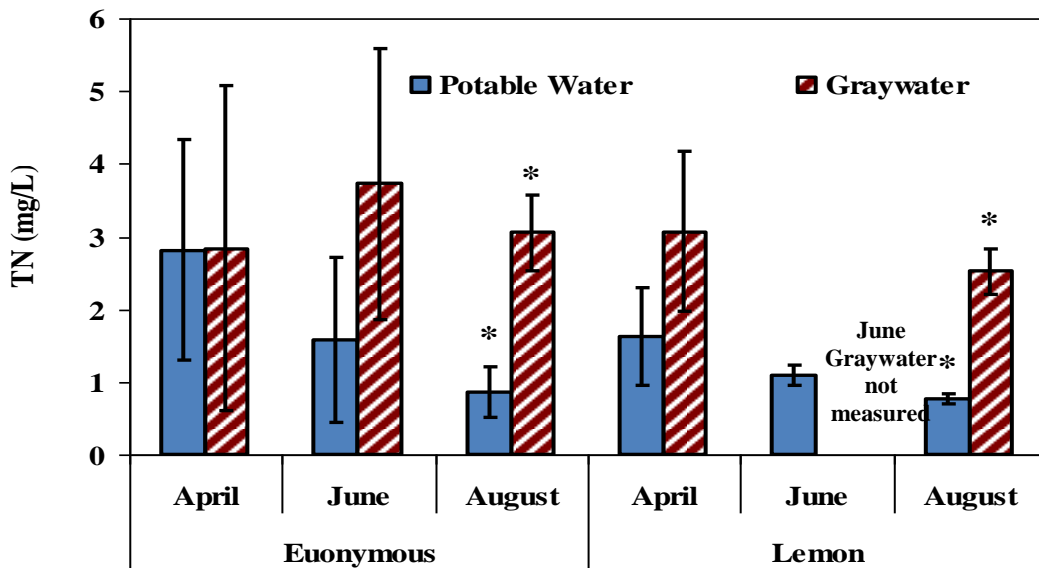


Figure 4.21 TN for Euonymous and Lemon (*synthetic graywater TN: 4.82 mg/L as N*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

The TN in the leachate from the graywater irrigated plants and grasses was less than the TN in the synthetic graywater (4.82 mg/L as N). For tall Fescue, in June and August, there was significant difference ($p < 0.05$) between the leachate from the potable water irrigated pots and the leachate from the graywater irrigated pots. Significant difference ($p < 0.05$) between the TN in the leachate from the potable water irrigated and the graywater irrigated pots for euonymous and lemon was found in August. The TN measured in the leachate from the graywater irrigated euonymous and lemon pots was higher than the TN measured in the leachate from graywater irrigated tall fescue and bermudagrass pots.

The TN was also measured for the case of irrigation of only soil (no plants or grasses; Figure 4.22). There was significant difference ($p < 0.05$) in the measured TN noted in June and August between the leachate generated from pots irrigated with graywater compared to those irrigated with potable water. In also comparing the TN in the leachate from the graywater irrigated pots with plants or grasses (on average between 1.1 mg/L to 3.7 mg/L) to the TN in the leachate from the graywater irrigated pots with only soil (no plants or grasses) (on average between 22 mg/L to 33 mg/L), it was found that there was more accumulation of TN when no plant or grass biomass was present to the point of substantial increase above the input TN concentration in the synthetic graywater (4.82 mg/L as N). It appears that the plant uptake of nitrogen plays an important role in the fate of nitrogen after irrigation.

It was determined that in April, for the graywater irrigated pots, on average, 70.4% of the TN was retained in the soil column for bermudagrass, with the percentage retained of TN for euonymous, lemon and tall fescue being respectively 41.1%, 36.4%

and 77.2% (Table 4.1). In June, for the graywater irrigated pots, the percentage retained for TN was 59.7%, 22.8%, and 77.8% for bermudagrass, euonymous, and tall fescue respectively. It was further observed that in June, similar to April there was more TN retained through the soil column (or taken up by roots) for grasses than for the plants. In August, for the graywater irrigated pots, for bermudagrass, euonymous, lemon and tall fescue, the percentage TN retained was on average 76.4%, 46.8%, 47.8% and 78.2% respectively.

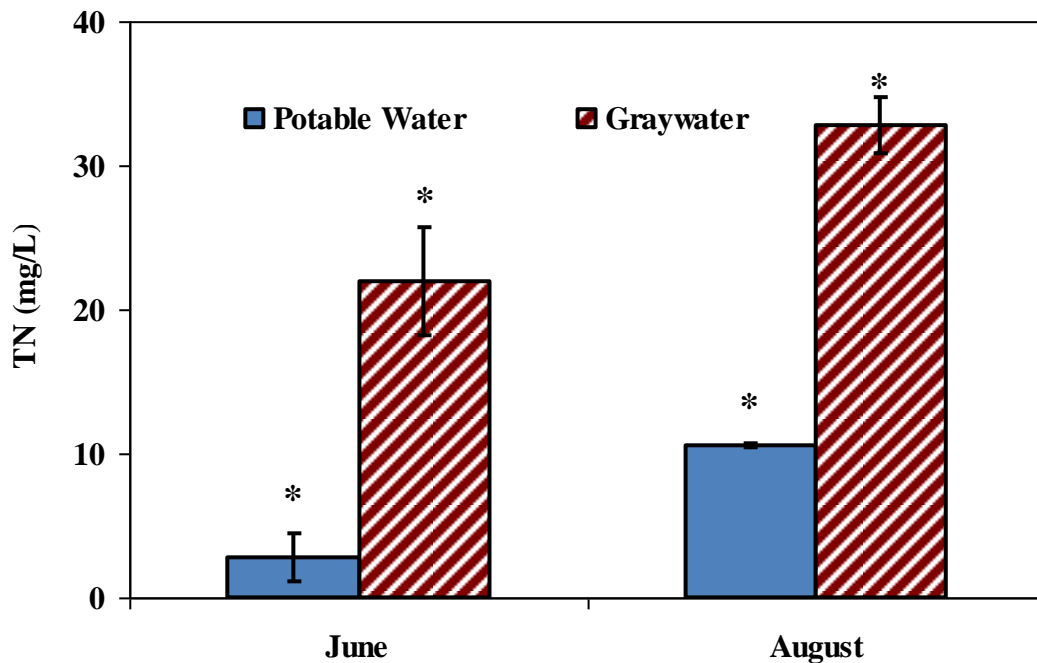


Figure 4.22 TN in Leachate Collected from Pots with No Plants (*synthetic graywater TN: 4.82 mg/L as N*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

4.8 Nitrate

In April, June and August, the nitrate measured in the leachate from the graywater irrigated pots was higher than the nitrate measured in the leachate from the potable water irrigated pots for bermudagrass, euonymous and lemon (Figures 4.23 and 4.24). For tall fescue, across the sampling periods, different trends were noted for the nitrate measured from the graywater irrigated pots compared to the potable water irrigated pots. In April and August, the nitrate measured in the leachate from the graywater irrigated tall fescue pots was lower than that measured in the leachate from the potable water irrigated tall fescue pots. However, in June, the nitrate measured in the leachate from the graywater irrigated and the potable water irrigated tall fescue pots was the same.

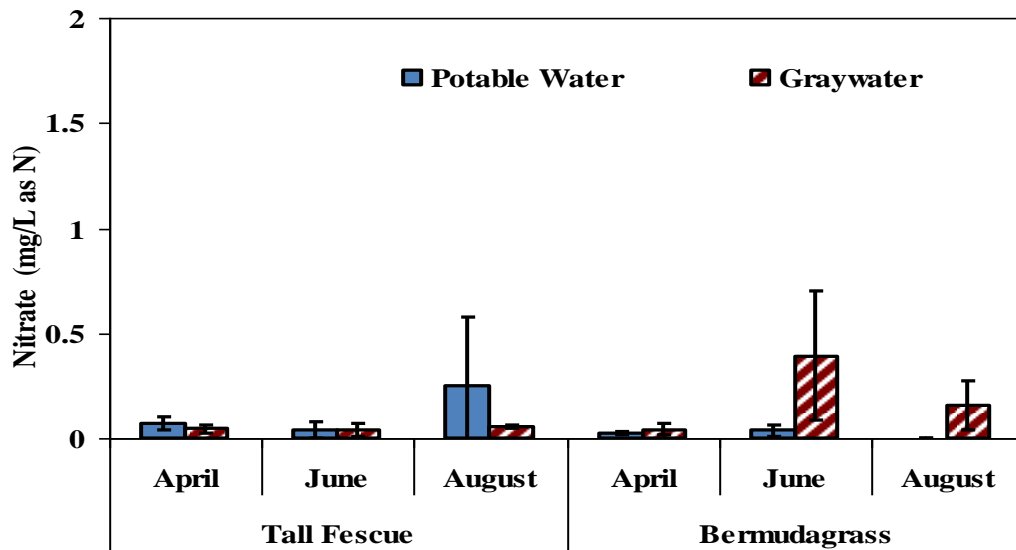


Figure 4.23 Nitrate for Tall Fescue and Bermudagrass (*synthetic graywater Nitrate: 2.60 mg/L as N*)

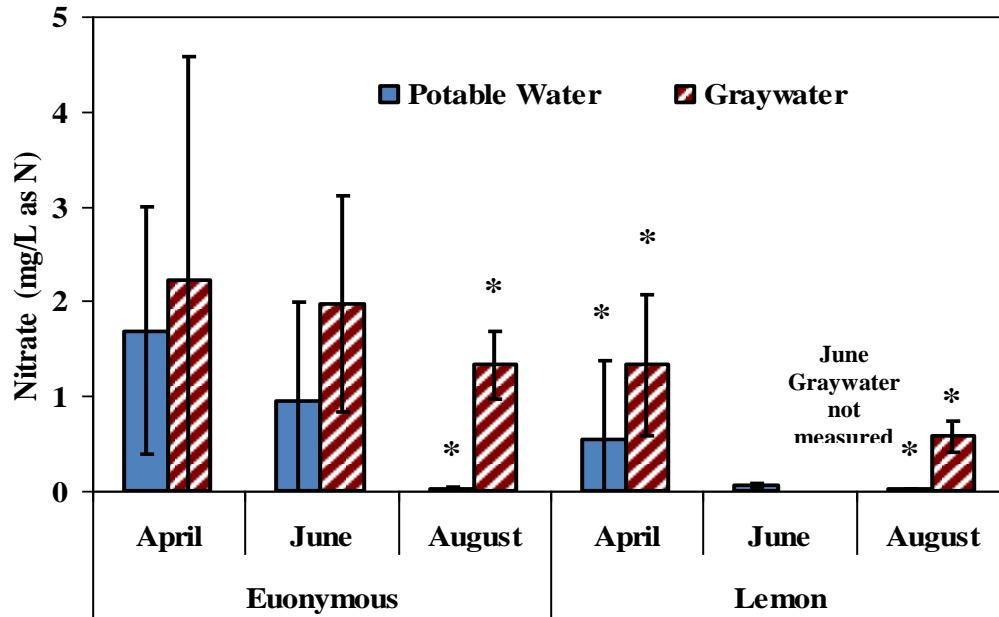


Figure 4.24 Nitrate for Euonymous and Lemon (*synthetic graywater Nitrate: 2.60 mg/L as N*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

The nitrate concentrations measured in the leachate for euonymous and lemon were on average higher than the nitrate concentrations measured in the leachate for tall fescue and bermudagrass in both cases where potable water and graywater were used for irrigation. It is possible that nitrate is not as readily metabolized by these plants as compared to the grasses studied. Significant difference ($p < 0.05$) was found between the nitrate concentrations in the leachate from the graywater and potable water irrigated euonymous pots in August. In April and August, significant difference ($p < 0.05$) was also determined for the nitrate in the leachate from the graywater and potable water irrigated lemon pots. Given that no statistically significant difference ($p > 0.05$) was determined across the three sampling events (no significant difference with time), the nitrate does not appear to accumulate with time in the graywater irrigated pots.

Nitrate concentrations measured for the graywater irrigated pots with only soil (no plants or grasses) (22 mg/L to 34 mg/L; Figure 4.25) were substantially higher than the nitrate measured in the leachate from the graywater irrigated pots containing plants or grasses (0.03 mg/L to 2.2 mg/L). This trend is in line with what was observed for the total nitrogen. In August, there was significant difference ($p < 0.05$) between the nitrate in the leachate from the potable water irrigated and the graywater irrigated pots with only soil. As was the case with the total nitrogen, the nitrate levels in the control pots (potable water irrigated) with only soil were higher than the nitrate measured in the leachate for the potable water irrigated tall fescue, bermudagrass, euonymous and lemon.

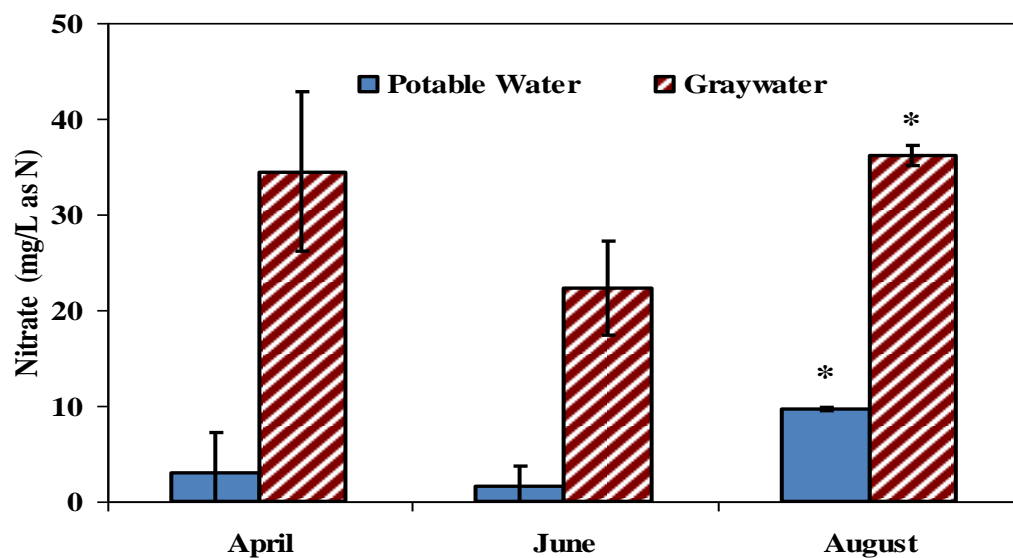


Figure 4.25 Nitrate in Leachate Collected from Pots with No Plants (*synthetic graywater Nitrate: 2.60 mg/L as N*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

4.9 Ammonium

For all three sampling events, the ammonium measured in the leachate from pots irrigated with graywater was higher than the ammonium measured in the leachate from pots irrigated with potable water for euonymous, tall fescue and lemon (Figures 4.26 and 4.27). However, none of the differences was statistically significant ($p > 0.05$). In June and August, the ammonium measured in the leachate from the graywater irrigated bermudagrass pots was higher than the ammonium measured in the leachate from the potable water irrigated bermudagrass pots. However, in April, the ammonium measured in the leachate from the graywater irrigated bermudagrass pots was only slightly different compared to the ammonium in the leachate from the potable water irrigated bermudagrass pots (0.09 mg/L (potable water) versus 0.08 mg/L (graywater)).

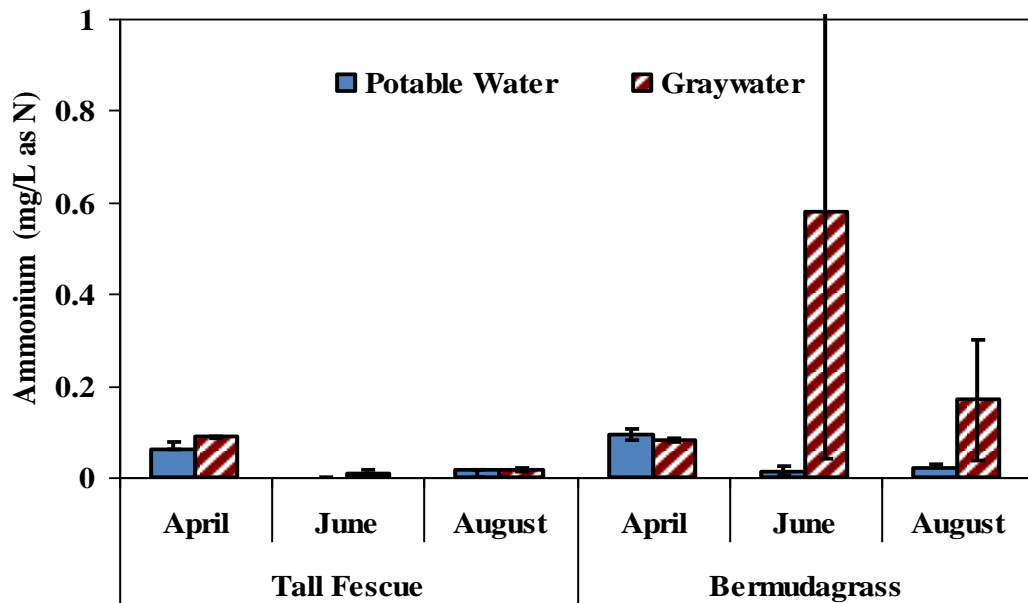


Figure 4.26 Ammonium for Tall Fescue and Bermudagrass (synthetic graywater Ammonium: 2.22 mg/L as N)

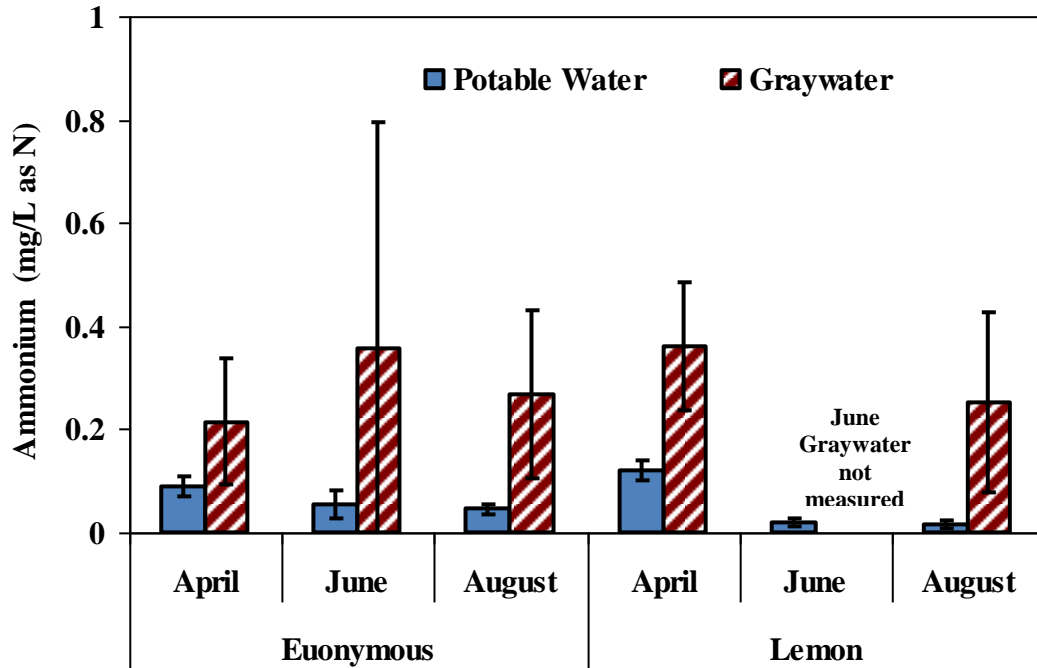


Figure 4.27 Ammonium for Euonymous and Lemon (*synthetic graywater Ammonium: 2.22 mg/L as N*)

The ammonium concentration in the leachate from all the graywater irrigated pots was lower than the ammonium in the synthetic graywater (2.22 mg/L as N) at all sampling events, indicating sorption of ammonium in the soil column or plant uptake (Figure 4.28). In April, the ammonium measured in the leachate from the graywater irrigated pots with only soil (no plants or grasses) was higher than the ammonium measured in the leachate from the graywater irrigated bermudagrass and tall fescue pots, but lower than the ammonium measured in the leachate from the graywater irrigated lemon and euonymous pots. However, in August, the ammonium measured in the leachate from the graywater irrigated pots with only soil was lower than the measured ammonium in the leachate from all the graywater irrigated plant and grass pots. The synthetic graywater contained very low levels of ammonium, and leaching of ammonium from pots irrigated with graywater does not appear to be a concern.

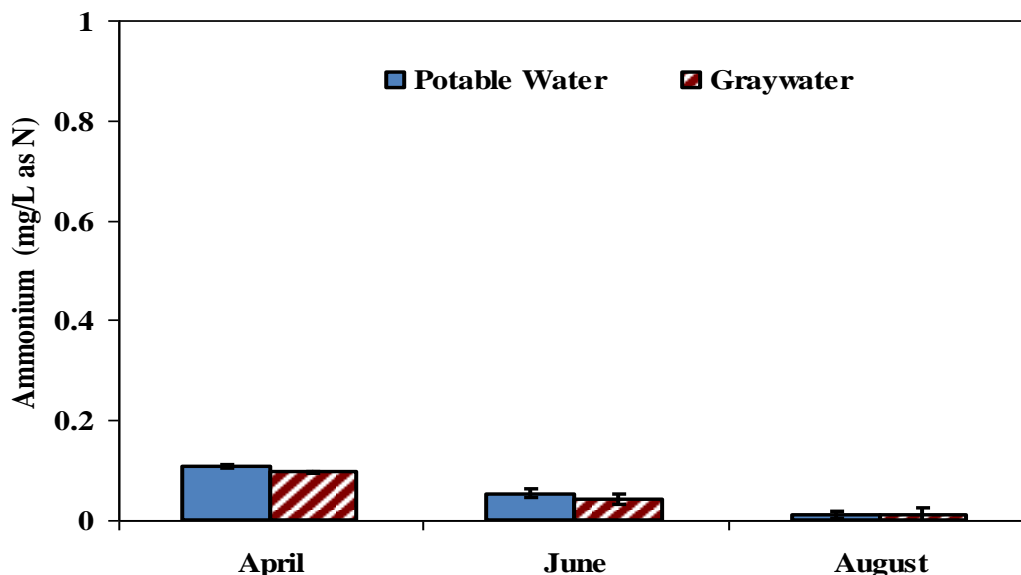


Figure 4.28 Ammonium in Leachate Collected from Pots with No Plants (*synthetic graywater Ammonium: 2.22 mg/L as N*)

4.10 Sodium Adsorption Ratio

Leachate analysis for SAR revealed that in April, June, and August, the SAR measured in the leachate from the graywater irrigated pots was higher than that measured in the leachate from the potable water irrigated pots for tall fescue, bermudagrass and lemon* (*in April and August; Figures 4.29 and 4.30). For euonymous, the SAR of the leachate from the graywater irrigated pots was higher than that measured in the leachate from the potable water irrigated pots in June and August. However, in April, for euonymous, the SAR of the leachate from the graywater irrigated pots was approximately the same as the SAR measured in the leachate from the potable water irrigated pots (SAR of 1.77 and 1.76 for the leachate from the potable water and graywater irrigated pots respectively).

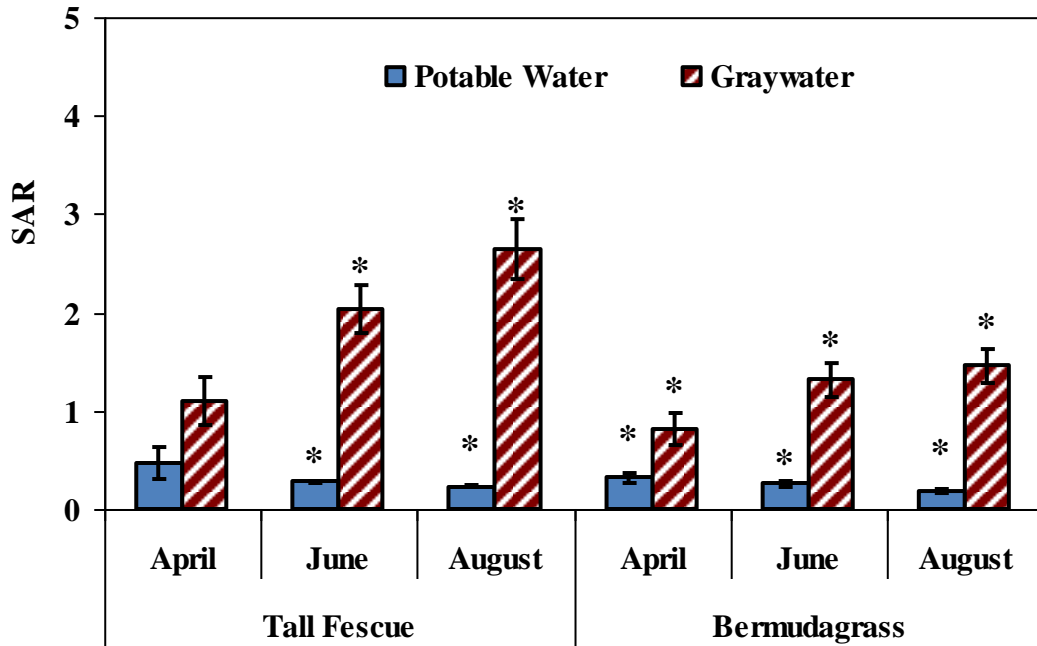


Figure 4.29 SAR for Tall Fescue and Bermudagrass (synthetic graywater SAR: 1.34) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

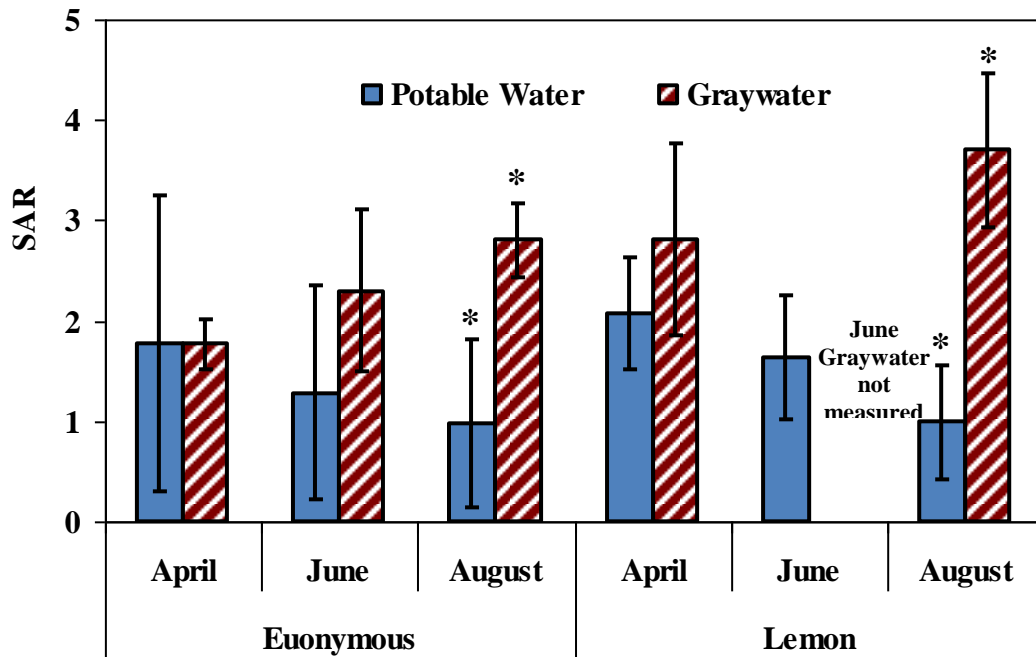


Figure 4.30 SAR for Euonymous and Lemon (synthetic graywater SAR: 1.34) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

For bermudagrass, in all three sampling months, significant difference ($p < 0.05$) was determined between the SAR measured in the leachate from the graywater irrigated pots and the SAR measured in the leachate from the potable water irrigated pots. Significant difference ($p < 0.05$) between the SAR of the leachate from the graywater irrigated and potable water irrigated pots was also found for tall fescue in June and August. For euonymous and lemon, significant difference ($p < 0.05$) between the SAR measured in the leachate from the graywater irrigated and the potable water irrigated pots was only found in August. A pattern that was observed was that with the exception of bermudagrass, which already had significant difference in April, the other plants and grasses progressed from having no significant difference ($p > 0.05$) between the SAR measured in the leachate from the graywater and the potable water irrigated pots in April to having significant difference in August ($p < 0.05$) between the SAR measured in the leachate from the pots irrigated with potable water and graywater.

It was further observed that the SAR of the leachate from the graywater irrigated pots increased from April to August for all the plants and grasses. It was determined in comparing the SAR increase across the months, that the difference for the SAR of the leachate from the graywater irrigated pots was significant ($p < 0.05$) for April to June, and April to August for bermudagrass and tall fescue. Bermudagrass also had significant difference ($p < 0.05$) for the SAR of the leachate generated from the graywater irrigated pots from June to August. For euonymous, significant difference ($p < 0.05$) was determined for the SAR of the leachate from the graywater irrigated pots from April to August. The elevated SAR levels for the leachate from the graywater irrigated pots compared to the potable water irrigated pots was expected given that the graywater is

contributing sodium, magnesium and calcium. The SAR further indicates that there is salt buildup in the soil with time.

In contrast to the trend observed for the SAR measured in the leachate from the graywater irrigated pots containing plants or grasses, for the graywater irrigated pots with no plants or grasses, the SAR measured in the leachate from the graywater irrigated pots was similar to the SAR measured in the leachate from the potable water irrigated pots (Figure 4.31). The SAR in April was 4.78 for both the leachate generated from the potable water irrigated and the graywater irrigated pots. In August, SAR values of 0.88 and 0.85 were measured for the leachate from the potable water irrigated and the graywater irrigated pots respectively. In June, the SAR for the leachate from the graywater irrigated pots was less than that from the potable water irrigated pots (1.90 in the leachate from the graywater irrigated pots versus 3.10 in the leachate from the potable water irrigated pots).

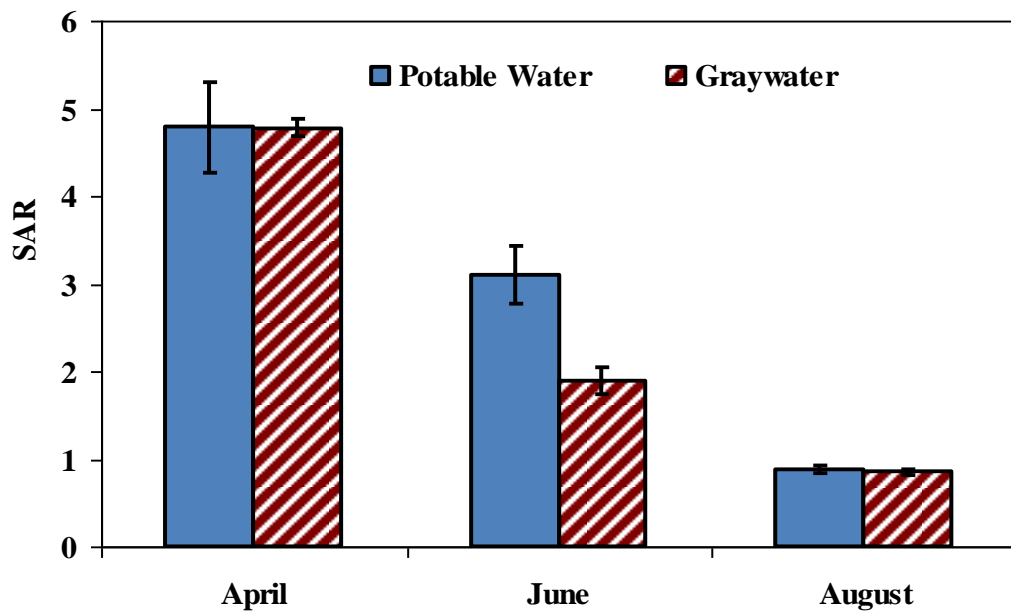


Figure 4.31 SAR in Leachate Collected from Pots with No Plants (*synthetic graywater SAR: 1.34*)

Two trends were noted in comparing the SAR measured in the leachate from the graywater irrigated pots with no plants or grasses to the SAR measured in the leachate from the graywater irrigated pots that contained plants or grasses. The first trend noted was that in April, the SAR measured for the leachate from the graywater irrigated pots with soil only (no plants or grasses – 4.78) was higher than the SAR measured in the leachate from the graywater irrigated pots that contained plants or grasses, with SAR values ranging from 0.81 in bermudagrass to 2.81 in lemon. By August, the reverse situation occurred such that the SAR in the leachate from the graywater irrigated pots with only soil (0.85) was lower than the SAR measured in the leachate from the graywater irrigated pots containing plants or grasses (1.45 to 3.70).

Secondly, the SAR measured in the leachate from the graywater irrigated pots with no plants or grasses decreased with time in contrast to the SAR measured in the leachate from the graywater irrigated pots containing plants or grasses. In April, high SAR was not only measured in the leachate from the graywater irrigated pots with no plants or grasses, but was also measured in the leachate from the potable water irrigated pots with only soil (no plants or grasses). This trend of high SAR measured in the control pots suggests that the high SAR measured in the leachate from the graywater irrigated pots with only soil cannot only be attributed to elevated SAR levels in the source graywater, but rather to the soil used in the pots as well.

Prior to the start of the experiments, the sandy loam soil used in the custom plant pots was found to have background levels of sodium, magnesium and calcium with a soil SAR of 4.0 (Chapter 3 – Section 3.3.2). The SAR measured in the leachate in April from both the graywater irrigated and the potable water irrigated pots was 4.78. Therefore, the

implication is that these cations (sodium, magnesium and calcium) were leaching out of the soil and were thus present in the soil leachate. Given that there is a finite concentration of sodium, calcium and magnesium in the soil, the SAR levels decreased with time. The trend noted in the SAR levels between the graywater irrigated pots with only soil and the graywater irrigated pots containing plants or grasses is an indication of the effect that plant or grass roots have on the SAR. With the plants or grasses, the sodium, calcium and magnesium are likely building up around the roots leading to elevated levels measured in the leachate. Alternatively, the presence of roots in the pots with plants or grasses could lead to the creation of migration pathways through the soil such that the cations are more readily leached out.

4.11 Conductivity

The conductivity measured in the leachate from the graywater irrigated pots (1090 $\mu\text{S}/\text{cm}$ to 5583 $\mu\text{S}/\text{cm}$) was found to be higher than the conductivity in the synthetic graywater (699 $\mu\text{S}/\text{cm}$). However, this higher conductivity in the graywater leachate compared to the graywater was expected, owing not only to the input of salts from the graywater but also from the cations already present in the soil, the particulates contributed from the plant and grass biomass that leach out after irrigation, and the subsequent presence of solids in the leachate. Therefore, it follows that there would also be elevated levels of dissolved solids in the leachate from the graywater irrigated pots which also account for the increased conductivity.

The conductivity measured in the leachate from the graywater irrigated pots was always higher than the conductivity measured in the leachate from the potable water

irrigated pots. It was further determined that for most of the sampling periods, the conductivity measured in the leachate from the graywater irrigated and the potable water irrigated pots were statistically significant ($p < 0.05$) (Figures 4.32 and 4.33). In particular, significant difference ($p < 0.05$) in the conductivity measured in the leachate from the graywater irrigated and the potable water irrigated pots was determined in June and August for tall fescue, in August for bermudagrass, and in April and August for both lemon and euonymous.

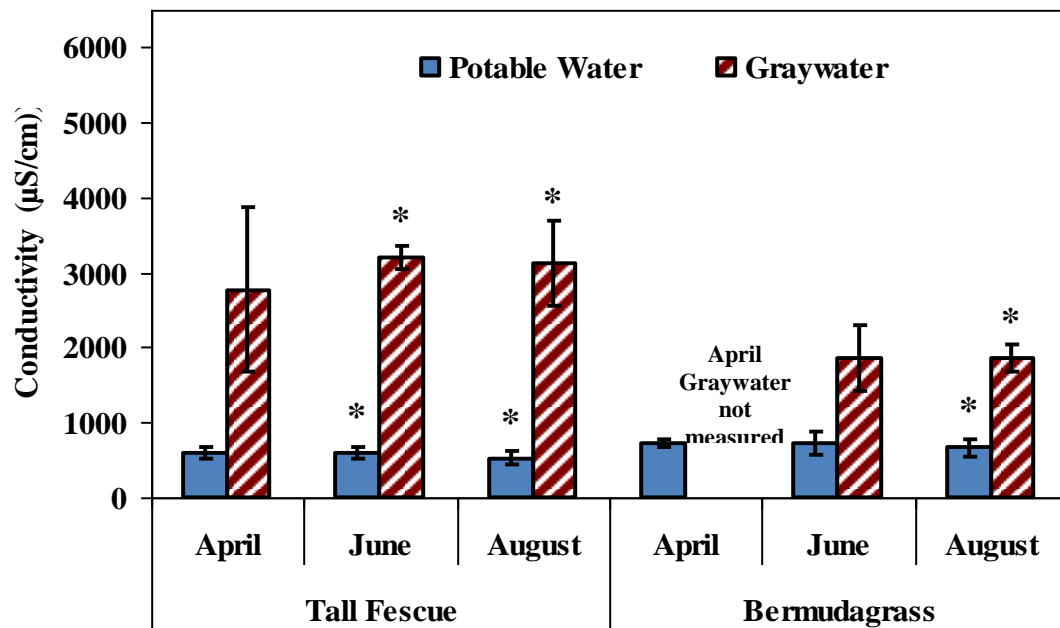


Figure 4.32 Conductivity for Tall Fescue and Bermudagrass (synthetic graywater conductivity: 699 $\mu\text{S}/\text{cm}$) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

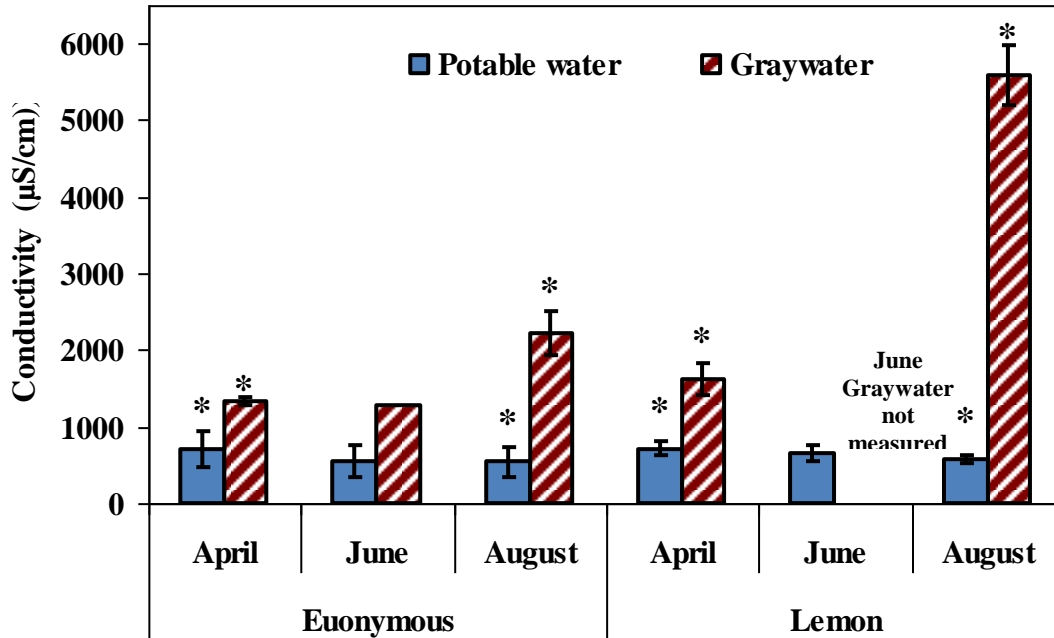


Figure 4.33 Conductivity for Euonymous and Lemon (synthetic graywater conductivity: 699 $\mu\text{S}/\text{cm}$) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

A distinct trend in the conductivity measured in the leachate from the graywater irrigated pots with time for tall fescue and bermudagrass between April to August was not found. The measured conductivity in the leachate from the graywater irrigated pots for tall fescue and bermudagrass was approximately the same across the sampling months. However, for lemon and euonymous, a significant difference ($p < 0.05$) with time between the conductivity measured in the leachate from graywater irrigated pots from April to August was found. The increase in the conductivity in the leachate from the graywater irrigated pots from April to August for the plants can be related to the uptake of salts around the root zone of plants that occurs. Given the deeper root zone of the plants compared to the grasses, the leaching potential of salts is also enhanced.

The conductivity in the leachate from the graywater irrigated pots with only soil (no plants) (1090 $\mu\text{S}/\text{cm}$ to 1313 $\mu\text{S}/\text{cm}$) was less than the conductivity measured in the

leachate from plants and grasses irrigated with graywater (1278 $\mu\text{S}/\text{cm}$ to 5583 $\mu\text{S}/\text{cm}$). The lower conductivity measured in the leachate from the graywater irrigated pots with no soil relates back to the effects that plant (or grass) roots have on the leaching potential of salts. In August, significant difference ($p < 0.05$) was determined between the conductivity in the leachate from the graywater irrigated pots and the potable water irrigated pots with only soil (no plants) (Figure 4.33).

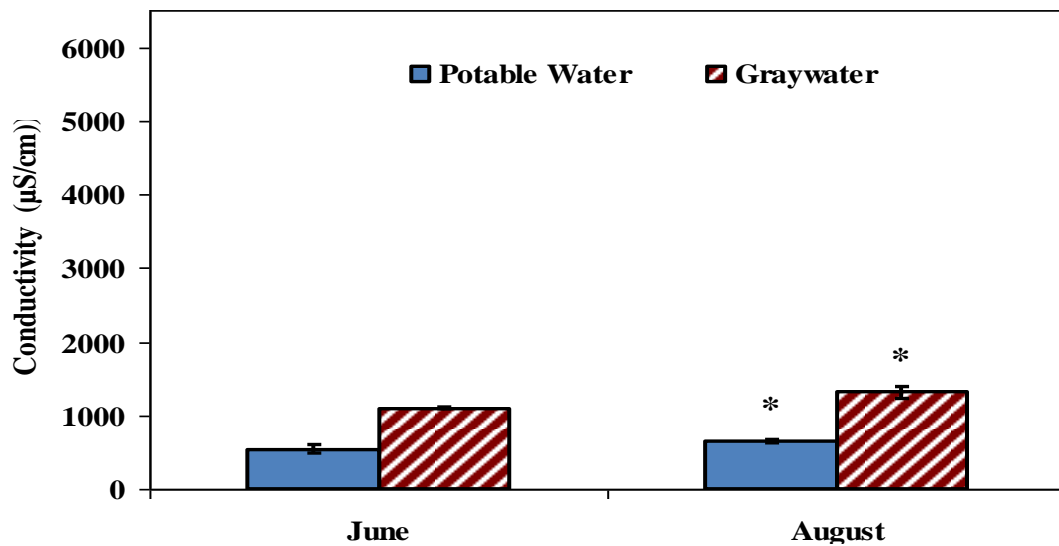


Figure 4.34 Conductivity in Leachate Collected from Pots with No Plants (*synthetic graywater conductivity: 699 $\mu\text{S}/\text{cm}$*) (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

4.12 Total Dissolved Solids

Consistent with the measured conductivity, the TDS was always higher in the leachate from the graywater irrigated pots compared to the leachate from the potable water irrigated pots (Figures 4.35 and 4.36). In August, for bermudagrass, tall fescue, lemon and euonymous, there was a significant difference ($p < 0.05$) between the TDS measured in the leachate from the graywater irrigated pots and the TDS measured in the

leachate from the potable water irrigated pots. Significant difference ($p < 0.05$) between the TDS measured in the leachate from the graywater irrigated and the potable water irrigated pots was also determined in April for tall fescue and euonymous, as well as in June for tall fescue. As was the case for the conductivity, it was found that the increase in TDS measured in the leachate from the graywater irrigated pots for euonymous between April to August was significant ($p < 0.05$).

The TDS measured in the leachate from the graywater irrigated pots was higher than the TDS measured in the leachate from the potable water irrigated pots for the pots containing only soil (no plants or grasses) (Figure 4.37). It was further determined that in the months of June and August, there was significant difference ($p < 0.05$) between the TDS measured in the leachate from the potable water irrigated pots and that measured in the leachate from the graywater irrigated pots. Similar to the trend noted for the conductivity, it was also noted that the TDS measured in the leachate from the soil only (no plants or grasses) pots irrigated with graywater (793 mg/L to 856 mg/L) was on average (825 mg/L) less than the TDS measured in the leachate from the graywater irrigated pots for tall fescue, bermudagrass, euonymous and lemon (850 mg/L to 3907 mg/L). Significant ($p < 0.05$) increase in the TDS measured in the leachate from the graywater irrigated pots with no plants or grasses occurred from June to August.

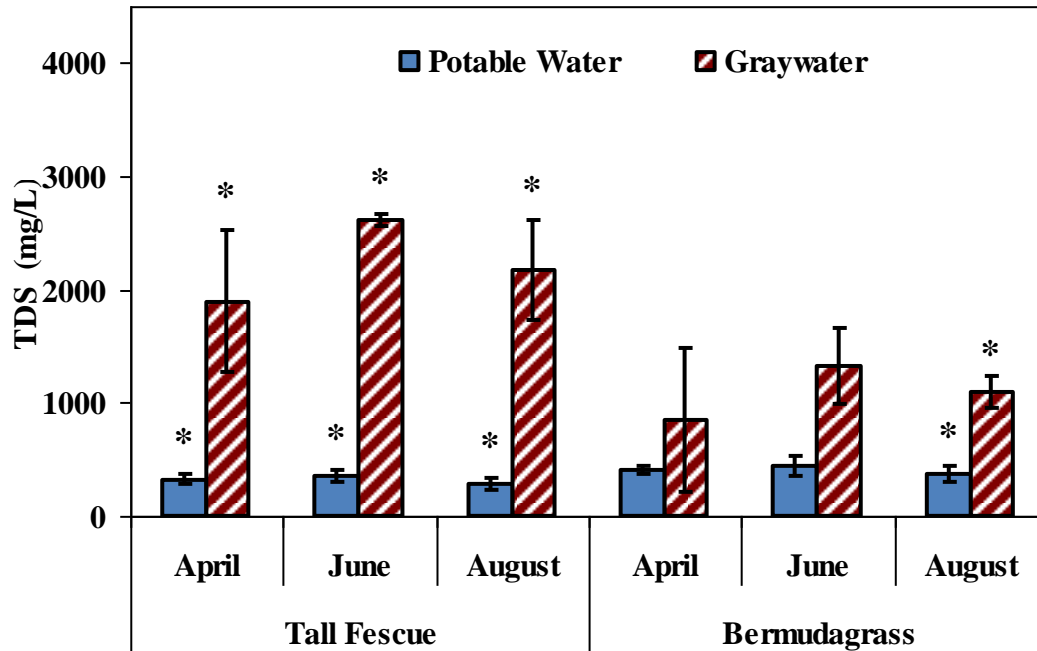


Figure 4.35 TDS for Tall Fescue and Bermudagrass (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

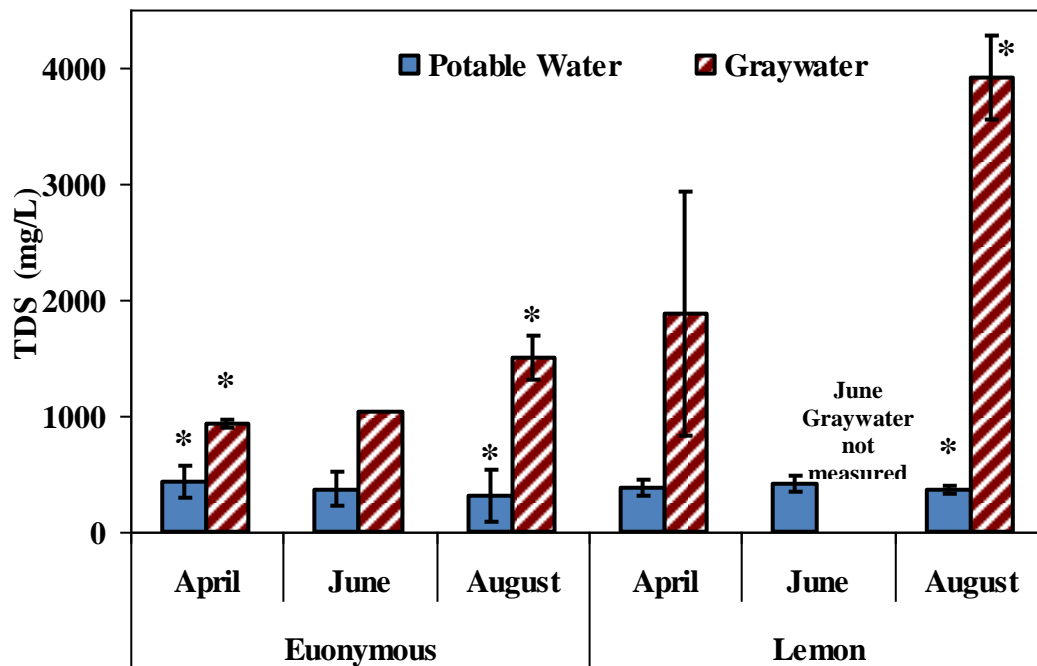


Figure 4.36 TDS for Euonymus and Lemon (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

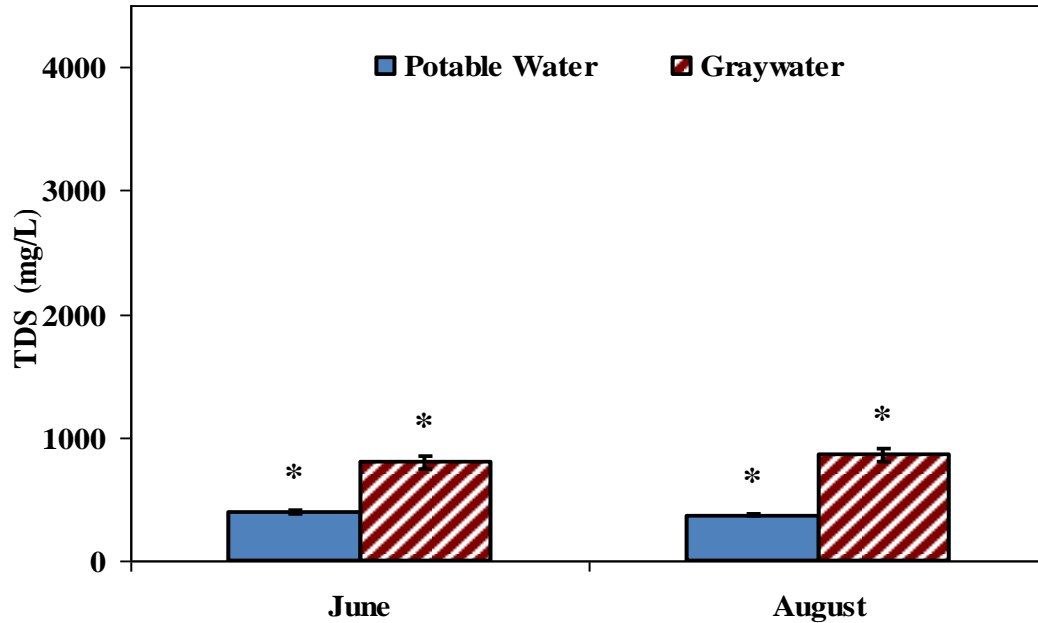


Figure 4.37 TDS in Leachate Collected from Pots with No Plants (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (June or August)

Conductivity measurements were well correlated to TDS measurements. Therefore, TDS measurements may be redundant and unnecessary for future sampling events. As previously discussed, for both the TDS and conductivity measured in the leachate from the graywater irrigated soil with no plants or grasses when compared to the measured TDS and conductivity in the leachate from graywater irrigated pots containing plants or grasses, an easily recognizable difference between these two scenarios (plants/grasses versus soil only) was ascertained. However, for the potable water irrigated pots, TDS and conductivity measured in the leachate from pots with no plants (366 mg/L to 396 mg/L and 539 $\mu\text{S}/\text{cm}$ to 642 $\mu\text{S}/\text{cm}$ for TDS and conductivity respectively) were similar to that measured in the leachate from the pots containing plants or grasses (283 mg/L to 440 mg/L and 519 $\mu\text{S}/\text{cm}$ to 730 $\mu\text{S}/\text{cm}$ respectively for TDS and conductivity). This implies that the substantially higher TDS and conductivity cannot solely be

attributed to contributions of particulates from the plants, but rather to mechanisms between the plant (or grass) biomass and the graywater constituents. Plant (or grass) root interactions with the graywater in soil results in the mobility of ions through the soil system.

4.13 Total Suspended Solids

The TSS measured in the leachate from pots irrigated with graywater was found to be higher than the TSS measured in the leachate from pots irrigated with potable water for tall fescue, bermudagrass, and lemon (Figures 4.38 and 4.39). Significant difference ($p < 0.05$) between the TSS measured in the leachate from graywater irrigated pots and the TSS measured in the leachate from the potable water irrigated pots was determined in all three sampling months for bermudagrass, but only in August for tall fescue and lemon.

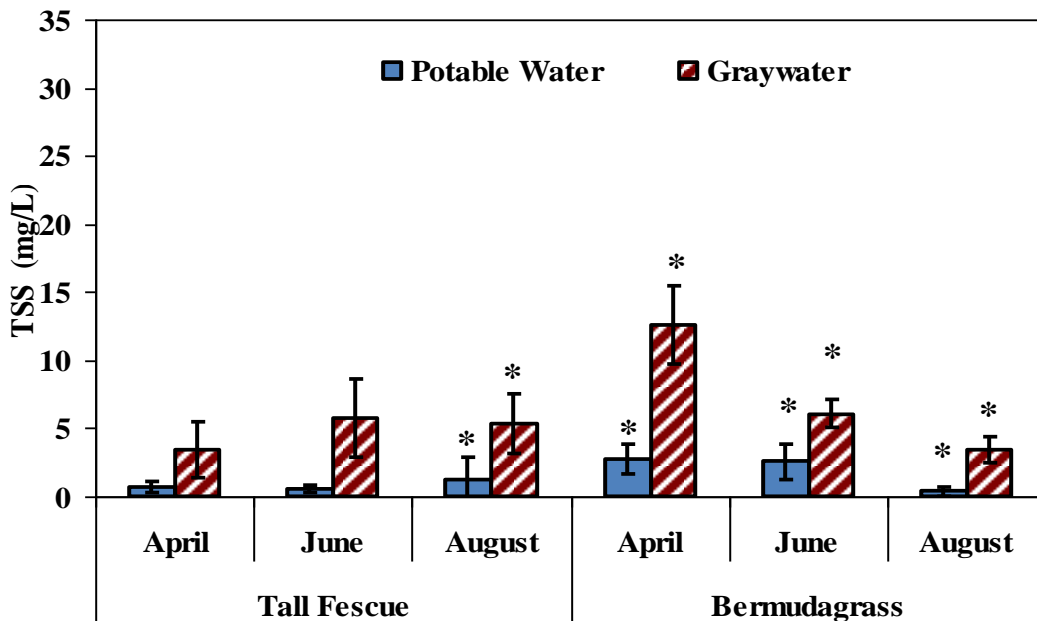


Figure 4.38 TSS for Tall Fescue and Bermudagrass (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

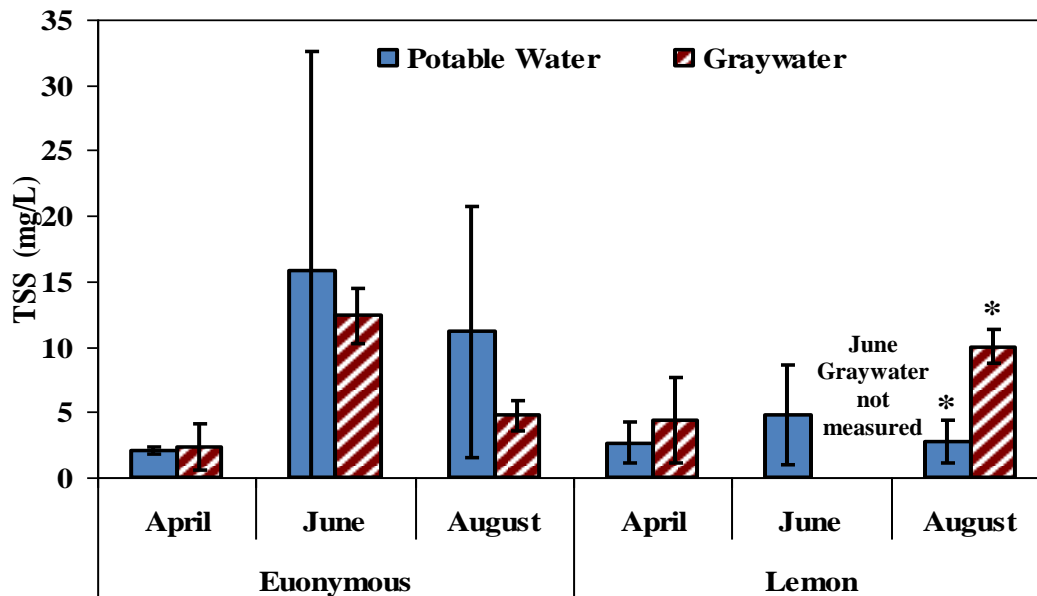


Figure 4.39 TSS for Euonymous and Lemon (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

Suspended solids are not only an indication of contaminant levels in a water system, but also serve as possible sorption sites for surfactants (Chapter 1 – Section 1.4.7.1). No discernible relationship between the TSS measured in the leachate from pots irrigated with graywater compared to measured LAS and AES concentrations in the leachate from the graywater irrigated pots was found. However, it was found that for the TSS measured in the leachate from the potable water irrigated pots containing plants or grasses, the highest TSS concentration measured was for euonymous in June, and the highest measured LAS concentration in the leachate from the potable water irrigated plant and grass pots was also for euonymous in June.

The TSS measured in the leachate from the graywater irrigated pots with only soil was higher than the TSS measured in the leachate from the potable water irrigated pots with only soil (no plants or grasses) (Figure 4.40). Similar to the results for the TDS, the

TSS in the leachate from the graywater irrigated pots with no plants or grasses (2.58 mg/L to 3.27 mg/L) was on average lower than the TSS in the leachate from the graywater irrigated pots with plants or grasses (2.31 mg/L to 12.58 mg/L).

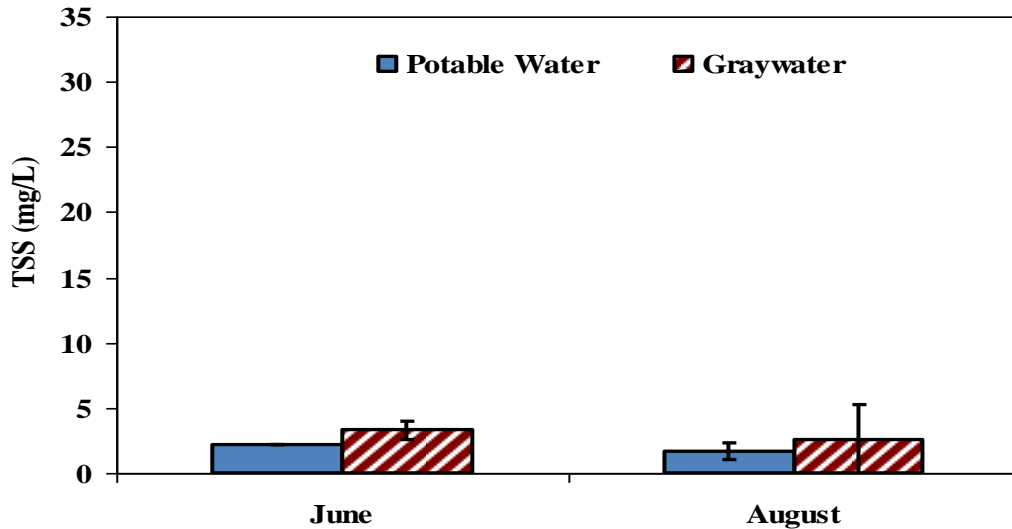


Figure 4.40 TSS in Leachate Collected from Pots with No Plants

4.14 Volatile Suspended Solids

VSS can serve as an indication of the possible presence of organics in a water system. The VSS measured in the leachate from pots irrigated with graywater was higher than the VSS measured in the leachate from pots irrigated with potable water (Figures 4.41 and 4.42). There was significant difference ($p < 0.05$) between the VSS measured in the leachate from pots irrigated with graywater and the VSS measured in the leachate from pots irrigated with potable water for tall fescue, bermudagrass and lemon in August. Significant difference ($p < 0.05$) between the measured VSS in the leachate generated from the graywater and potable water irrigated pots was also determined for tall fescue in June. A discernable trend for the VSS measured in the leachate from the graywater irrigated pots with time was not found.

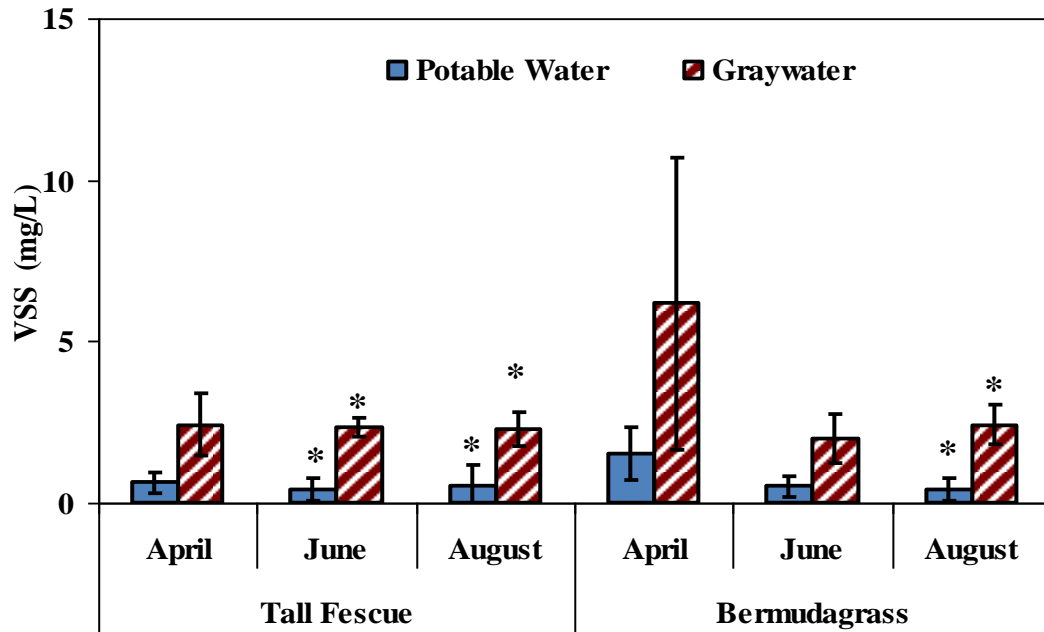


Figure 4.41 VSS for Tall Fescue and Bermudagrass (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

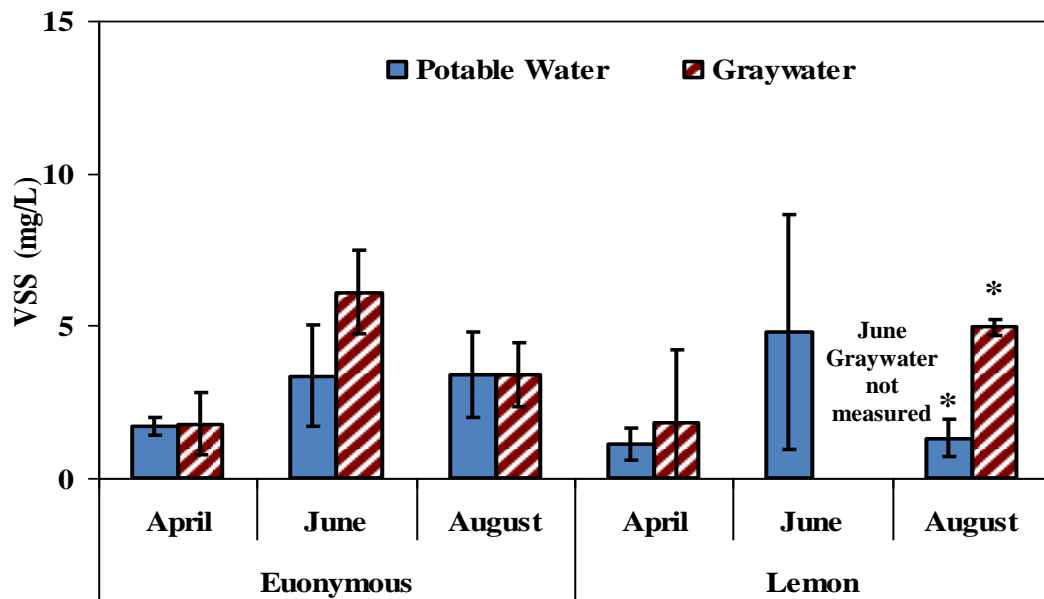


Figure 4.42 VSS for Euonymous and Lemon (*) indicates observance of a significant difference ($p < 0.05$) between graywater and potable water irrigated for a single sampling event (April, June or August)

Based on the results obtained for the VSS in August, it was observed that there was an error in the obtained data for the VSS measured in the pots containing only soil (no plants or grasses) irrigated with graywater or potable water. In June, the average VSS in the leachate from the potable water irrigated soil with no plants (0 mg/L) was less than the VSS measured in the leachate from the graywater irrigated soil with no plants (on average 1.33 mg/L) (data not shown).

4.15 Visual Plant Monitoring

The plants and grasses used were monitored visually throughout the experiment duration (Figures 4.43 to 4.46 - photographs of the plants and grasses as of September 2010). It was observed that for the graywater irrigated grasses, the grass color had a distinctive darker green color compared to the potable water irrigated grasses. Growth of the graywater irrigated grasses was also more rapid than the potable water irrigated grasses. The leaves of the graywater irrigated lemons were also greener than the leaves of the potable water irrigated lemons, though both the graywater and potable water irrigated lemon showed signs of chlorosis.

Though these visual observations of plant health were made, conclusions cannot be made regarding the impact of graywater to plant health. To arrive at a scientifically based conclusion, extensive tests such as determining die back from the tip and measuring leaf size would have to be performed. For the dwarf citrus and euonymus, plant health can be assessed based on dieback from the tip, foliage color (chlorosis), crown density, as well as the presence of insects and disease. For the grasses, quality can be assessed based on shoot density, uniformity, canopy color and the presence of weeds,

insect and disease. For both plants, observations of the plant roots can be made. Growth can be determined by measuring the height and diameter of the plant. The leaf size can be determined by using a LI-COR 3100 leaf area meter. The number of blooms on representative plants can be counted to determine the overall percent bloom.



Figure 4.43 *Left to Right:* Side-by-Side comparison of graywater and potable water irrigated Bermudagrass; Potable water irrigated Bermudagrass; Graywater irrigated Bermudagrass



Figure 4.44 *Left to Right:* Side-by-Side comparison of graywater and potable water irrigated Tall Fescue; Potable water irrigated Tall Fescue; Graywater irrigated Tall Fescue



Figure 4.45 *Left to Right:* Potable water irrigated Euonymous; Graywater irrigated Euonymous



Figure 4.46 *Left to Right:* Potable water irrigated Lemon; Potable water irrigated Lemon; Graywater irrigated Lemon; Graywater irrigated Lemon

In addition to the previously mentioned tests, to further determine the accumulation of graywater constituents, tissue analysis would have to be conducted. This tissue analysis would have to be carried out at the end of the experiment, in order to quantify the sodium, chloride, boron, potassium, magnesium, phosphorus, nitrogen and chloride content.

4.16 Summary

Based on the reviewed literature on measured graywater constituent concentrations, behavior of such constituents such as the sorption and biodegradation of surfactants, a set of hypotheses were developed (Chapter 1 – Section 1.6) regarding the fate of graywater constituents in this study. These hypotheses are again reviewed in this section:

Given the leaching behavior of nitrogen, it is expected that concentrations of this nutrient measured in the leachate from graywater irrigated soils will be elevated compared to nitrogen measurements in control scenarios where leachate from potable water irrigated soils are measured. However, given the removal mechanisms that occur as a result of plant uptake, the nitrogen measured in the leachate from graywater irrigated soils will be less than the measured nitrogen concentration in the graywater. For cases where plants are not present, and there is therefore the elimination of nutrient uptake by plants, higher levels of nitrogen will be measured in the leachate from soils with no plants compared to cases where plants are present.

Boron accumulation has been determined in graywater irrigated soil. It is therefore expected that boron will also accumulate in the graywater irrigated soils in this study. Therefore, the boron measured in the soil leachate will indicate accumulation of boron in the soil with time.

With high contributions of sodium from graywater, along with salt contributions, as is typically the case in residences, it is expected that elevated salt levels and dissolved solids will be detected in measured leachate concentrations. Elevated EC is also expected

given the effects that graywater can have on soil alkalinity and the increased salt content in the soil that will occur as a result of graywater irrigation.

Based on the sorption and biodegradation behavior of LAS, AE and AES, it can be stipulated that these surfactants will have lower concentrations in the soil leachate compared to the concentrations in the graywater. LAS use is typically higher compared to AES and AE (Chapter 1 - Section 1.4.5.2) and this leads to higher LAS concentrations often measured in the field (Chapter 2 – Section 2.3.3.1). Given that simulated conditions in this study will consist of higher LAS concentrations relative to AES and AE, coupled with the use of sandy loam soil in this study, it is believed that higher LAS will be measured in the leachate compared to AES and AE.

In assessing the leachate analysis results, trends noted were reviewed and compared to determine whether the expected trends stipulated in the above hypotheses occurred. In considering specific trends observed during the greenhouse experiments for the TOC, it was observed that between the months of April and August, there was accumulation of organic carbon, which was higher in the leachate from the graywater irrigated pots compared to the leachate from the potable water irrigated pots. The higher contribution of organic carbon can be attributed to the higher organics in the synthetic graywater which is contributed from the surfactants and yeast extract. Higher organic content in the leachate will also suggest that the organic content in the soil has elevated levels as well. The significance of a higher organic content is associated with the potential impact on the sorption behavior of surfactants (Chapter 1 – Section 1.4.7.1). Despite the elevated levels of TOC measured in the graywater, the average measured

TOC concentration for leachate from the graywater irrigated plant and grass pots (19 mg/L) was still below the TOC for the synthetic graywater (84.91 mg/L).

The LAS concentrations measured in the leachate from the graywater irrigated pots were higher than the measured AES concentrations in the leachate from the graywater irrigated pots. This trend can be attributed to higher input concentrations of LAS in the graywater and lower LAS sorption mechanisms. Also, the higher concentrations of LAS measured in the leachate from the graywater irrigated pots containing plants or grasses compared to the graywater irrigated pots with only soil implies that there is less sorption of LAS to the soil when plants or grasses are present, but more sorption to soil in the absence of plant or grass biomass.

For Boron, it was observed that in June and August, there was accumulation above the input boron concentration in the graywater. This boron accumulation is in line with what has been found in literature studies (Shafran et al. 2006, Gross et al. 2005). In countries where boron is included in laundry detergents, and in Western states where boron can be found in soil, boron accumulation would be a concern.

For total nitrogen, it was observed that between the months of April to August, the leachate from the graywater irrigated pots had increasing concentrations of total nitrogen. However, the nitrogen in the leachate from the graywater irrigated pots containing plants or grasses was still below the input nitrogen concentration in the graywater. The implication is that with time, build up of nitrogen will occur. The total nitrogen measured in the leachate from the graywater irrigated pots containing soil only (no plants or grasses) was substantially higher than the total nitrogen measured in the leachate from the graywater irrigated pots containing plants or grasses and was

subsequently above the input graywater total nitrogen concentration. This is consistent with the stated hypothesis for nutrients and illustrates the impact plants and grasses have on nitrogen uptake. However, of note is that the leachate measured from the control pots (potable water irrigated) also had elevated levels of nitrogen. Nitrogen levels cannot only be attributed to the graywater but also to background concentrations in the soil (Chapter 3 – Section 3.3.2). In comparing the total nitrogen, ammonium and nitrate between the plants and grasses, it was found that for the plants, the total nitrogen, and subsequently the nitrate and ammonium measured in the leachate from the graywater irrigated plants was higher than that measured in the leachate from the graywater irrigated grasses. This suggests that the plant and grass root impact the uptake of nitrogen, with more uptake occurring in the grasses.

As was expected, the SAR measured in the leachate from the graywater irrigated pots was higher than the SAR measured in the leachate from the potable water irrigated pots. The SAR values also increased with time, thus implying that there is salt buildup. The SAR can impact the infiltration capacity of the soil and thus affect the leachate generation capability. Impacts of the SAR and surfactants could play a role in the reduced leachate concentration measured from the graywater irrigated pots compared to potable water irrigated pots.

As expected, there was a similar trend noted between the TDS and conductivity in the leachate from the graywater irrigated pots. The measured TDS concentrations as reported in the leachate from the graywater irrigated pots were significantly higher ($p < 0.05$) than the concentrations measured in the leachate from the potable water irrigated pots. This suggests that possible salt buildup is occurring in the soil.

Analysis of the results revealed that even for the control pots, for some measured parameters, statistically significant differences ($p < 0.05$) occurred in sampling events over time. This implies that the background concentrations of nutrients and other soil parameters associated with the soil have an effect on concentrations measured in the graywater and potable water leachate given that leachate results include contributions from soil constituents that are leaching from the soil through the depth of the soil profile. However, as previously noted, the concentrations measured in leachate from the graywater irrigated pots were higher than those in the leachate from the potable water irrigated pots.

Nutrient and surfactant accumulation in the soil still have to be assessed. This will be carried out after the last leachate sample is collected. It is clear that plant presence and type can have substantial impact on the fate of graywater constituents. Soil analysis is required in order to determine whether graywater constituents are accumulating in the soil or degrading downgradient through the soil profile. Though plant roots impact the fate of surfactants, surfactant removal occurs and of noteworthy concern is that surfactants still penetrate the soil profile and can cause problems in areas where the groundwater table is low.

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APPENDIX A

LEACHATE GENERATION AND ANALYSIS

RESULTS FOR

INDIVIDUAL PLANT AND GRASS POTS

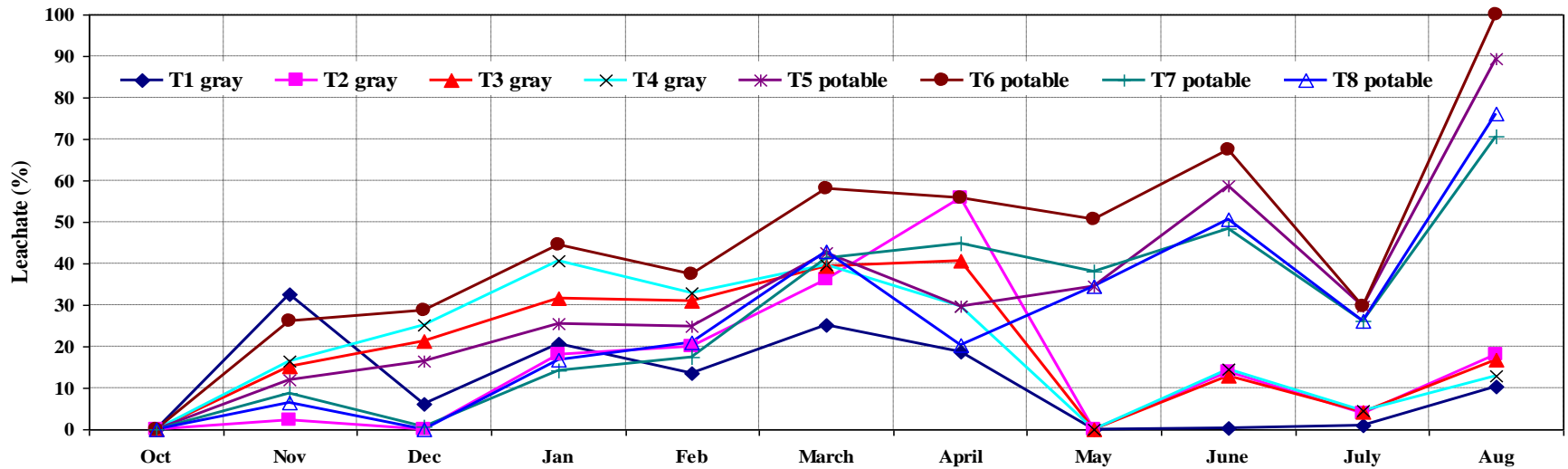


Figure A.1 Leachate Tall Fescue

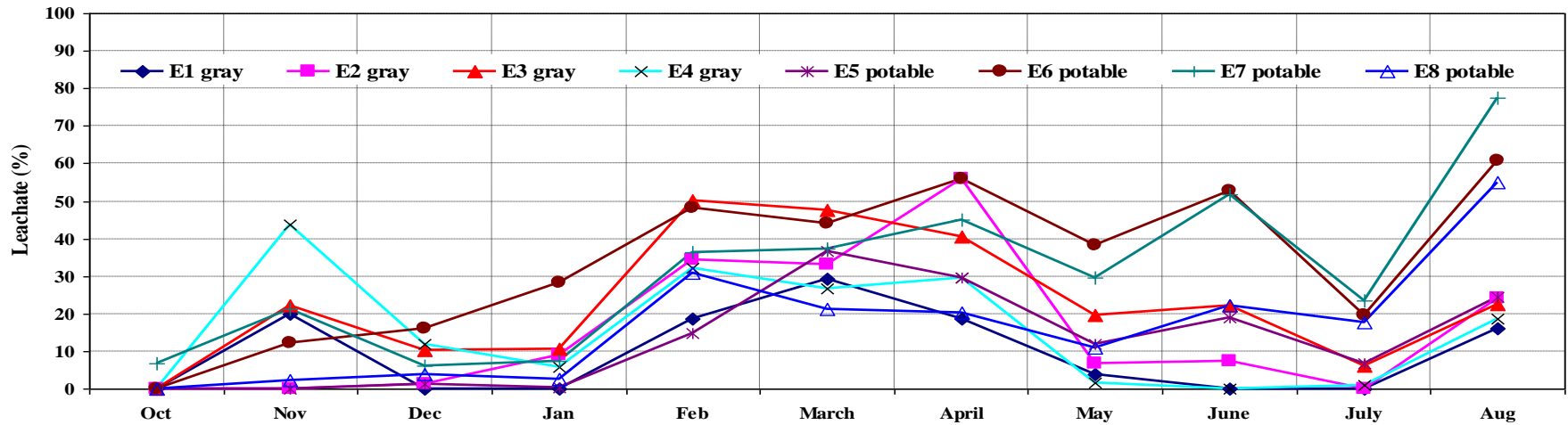


Figure A.2 Leachate Euonymous

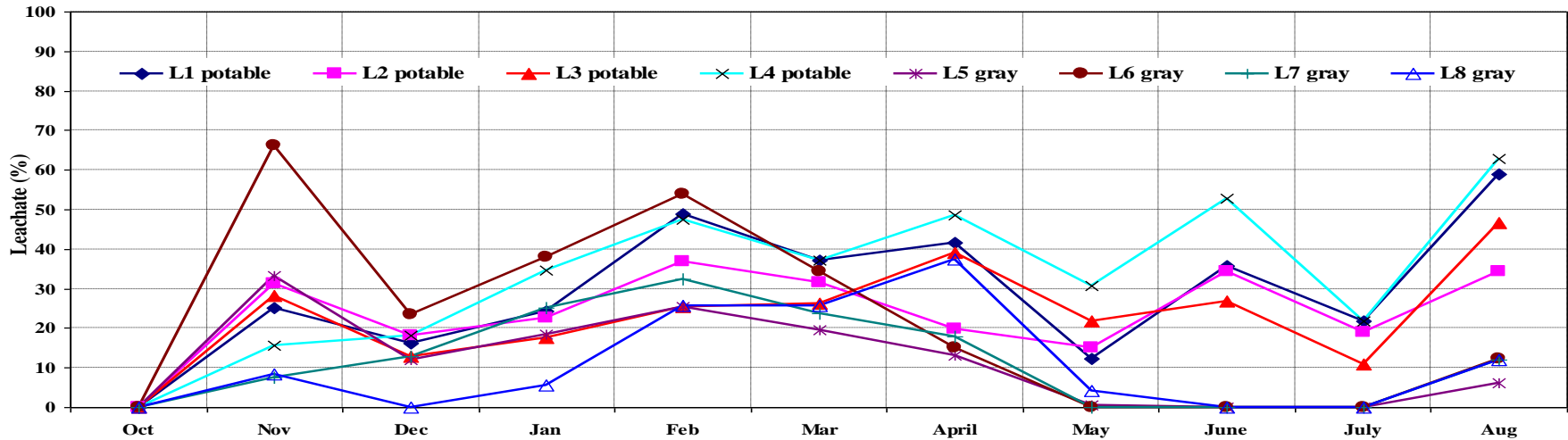


Figure A.3 Leachate Lemon

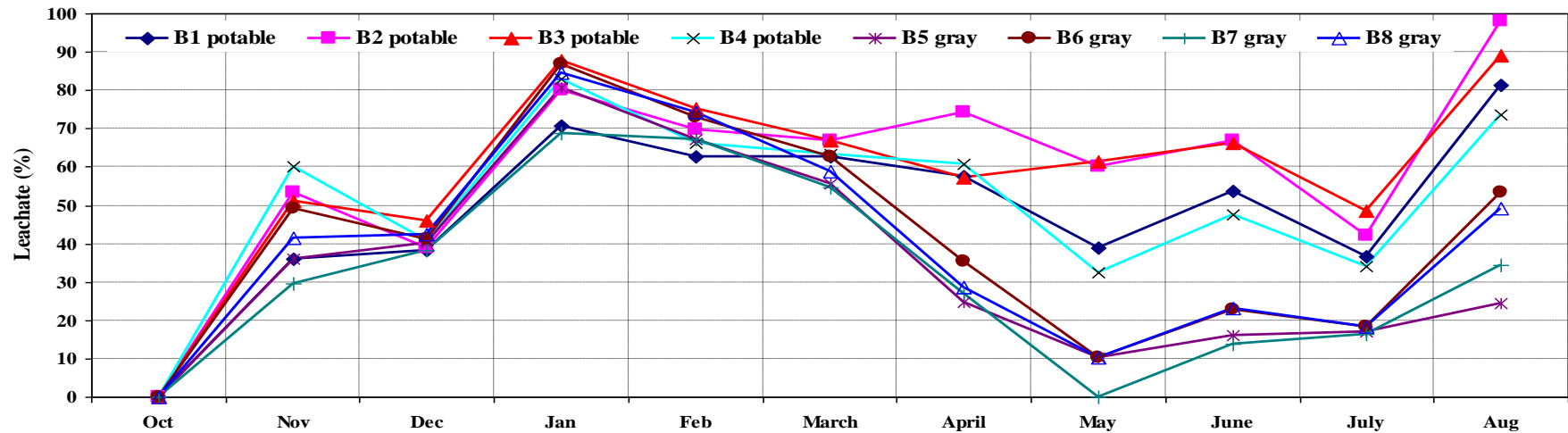


Figure A.4 Leachate Bermudagrass

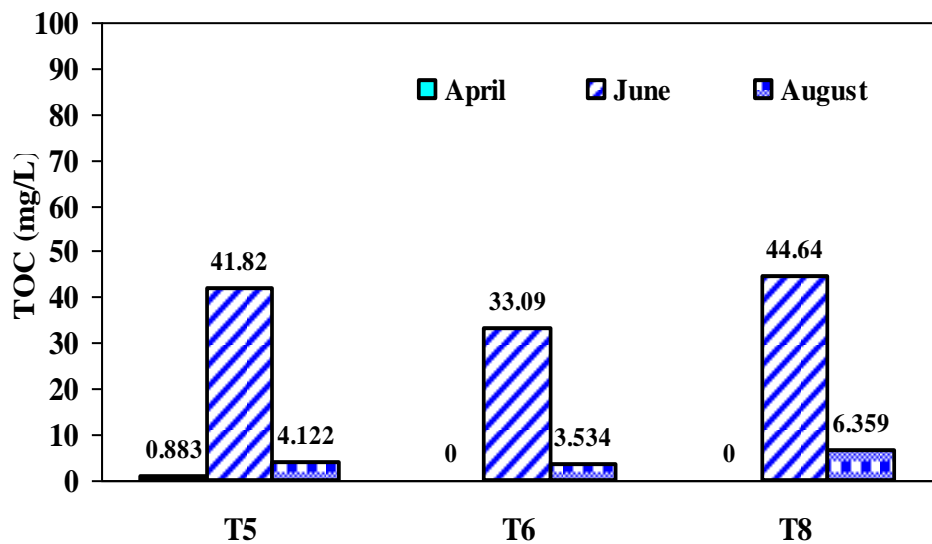


Figure A.5 TOC-Tall Fescue Potable Water

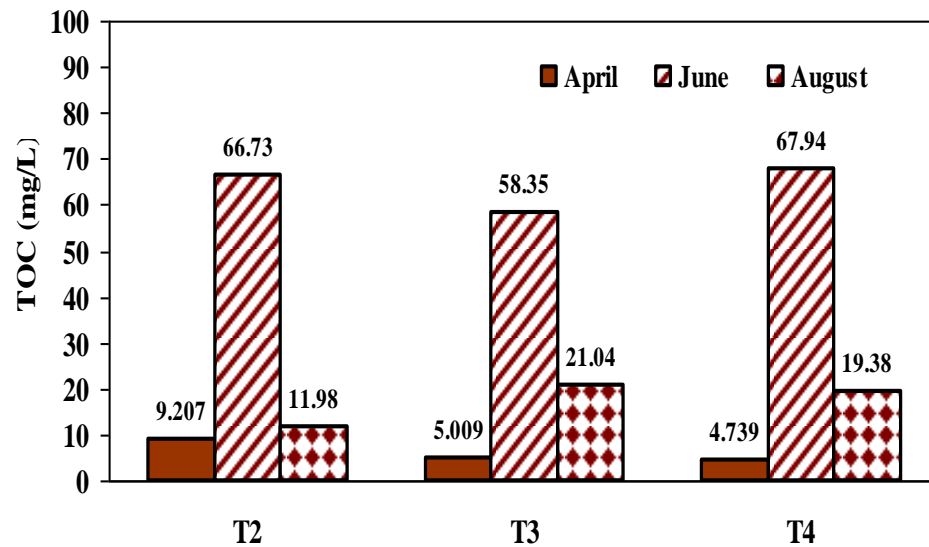


Figure A.6 TOC-Tall Fescue Graywater

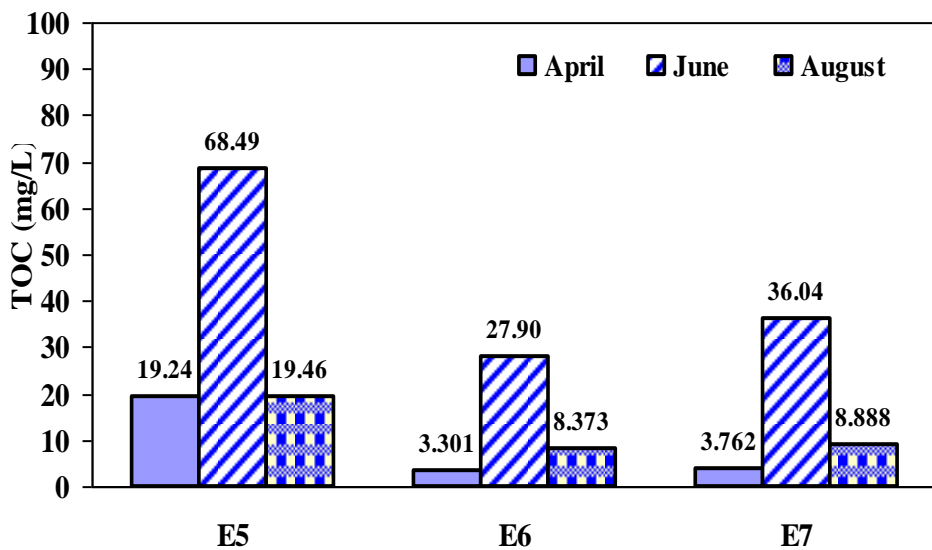


Figure A.7 TOC- Euonymous Potable Water

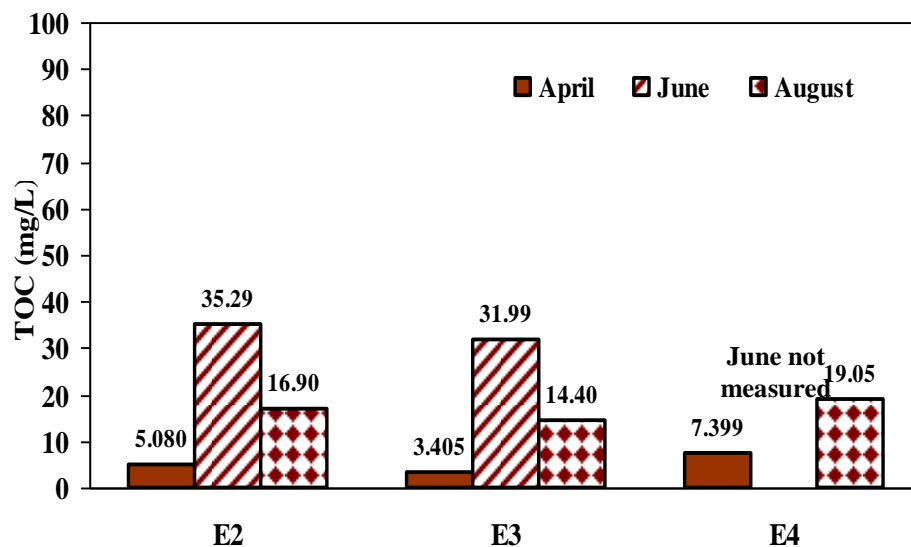


Figure A.8 TOC- Euonymous Graywater

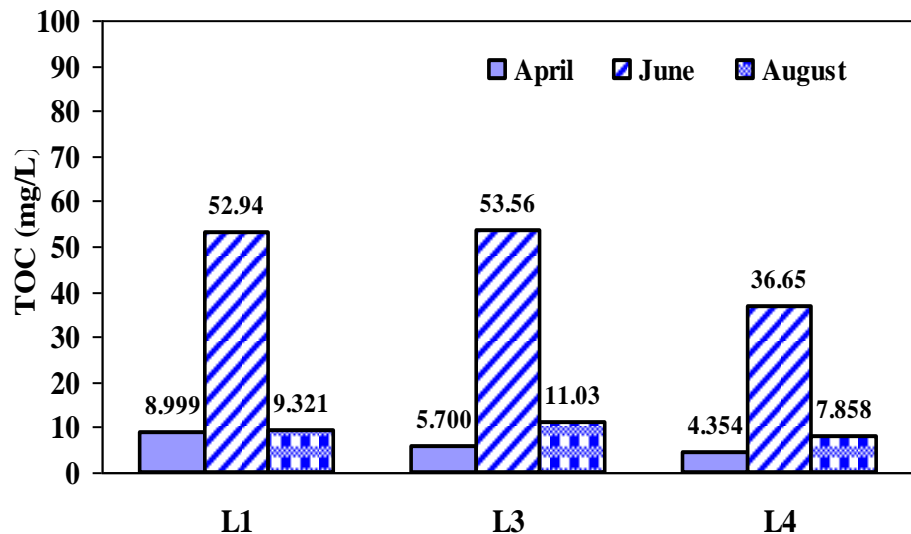


Figure A.9 TOC- Lemon Potable Water

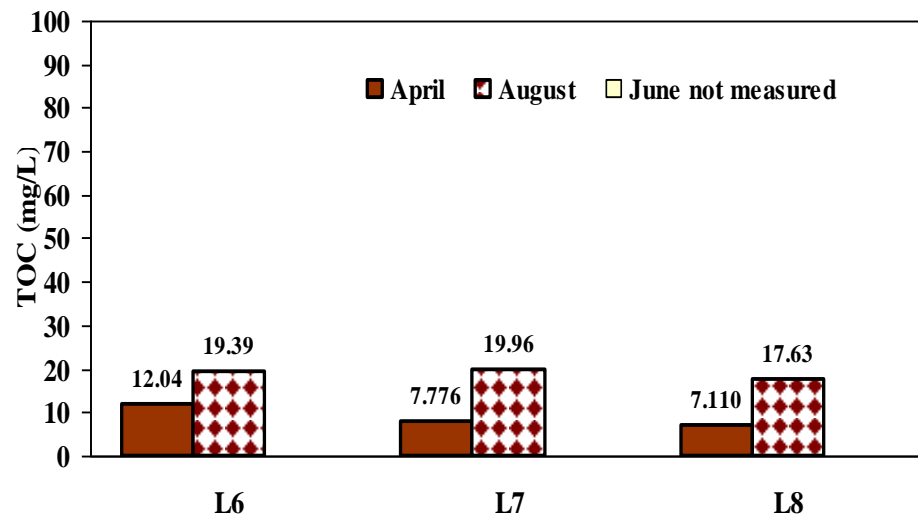


Figure A.10 TOC- Lemon Graywater

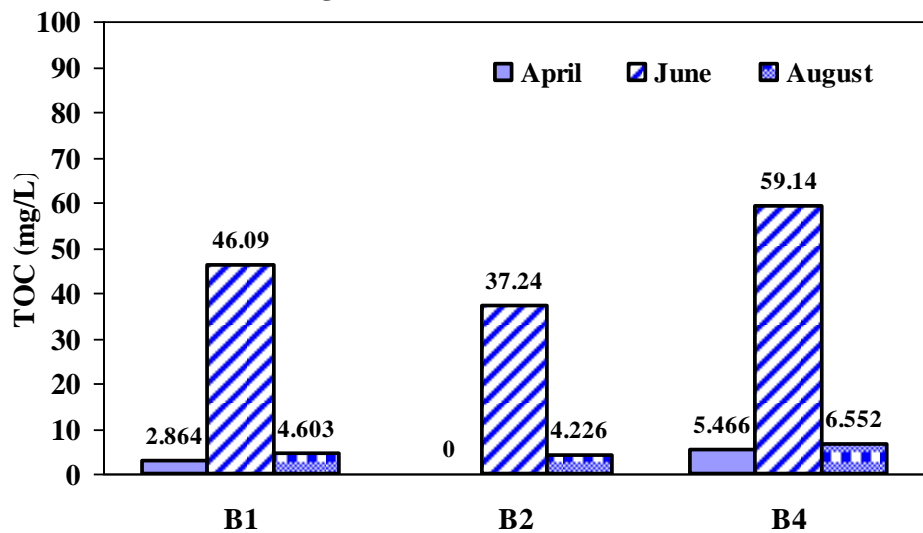


Figure A.11 TOC- Bermudagrass Potable Water

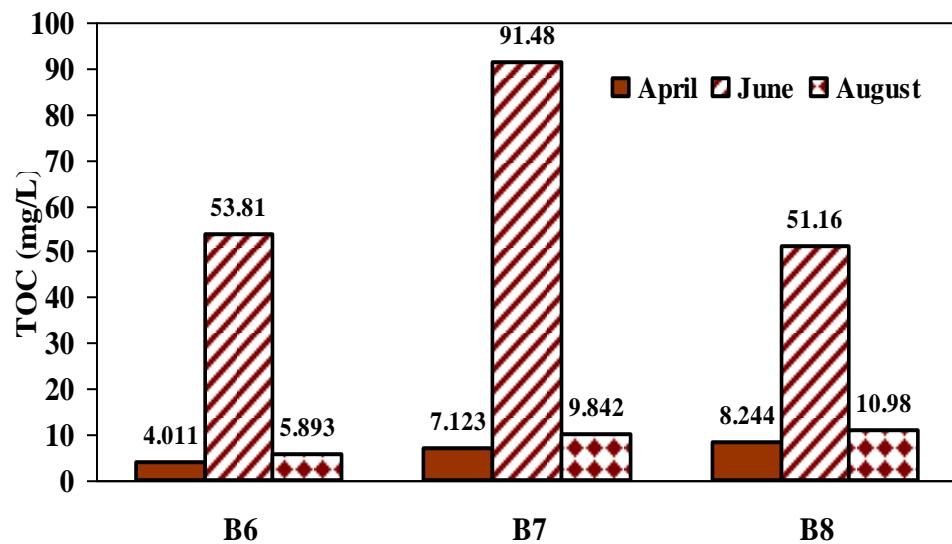


Figure A.12 TOC- Bermudagrass Graywater

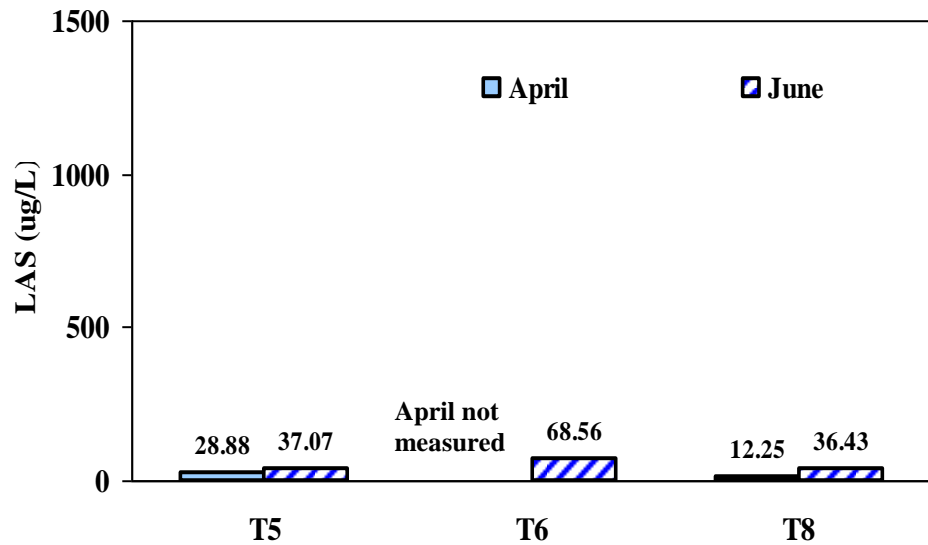


Figure A.13 LAS-Tall Fescue Potable Water

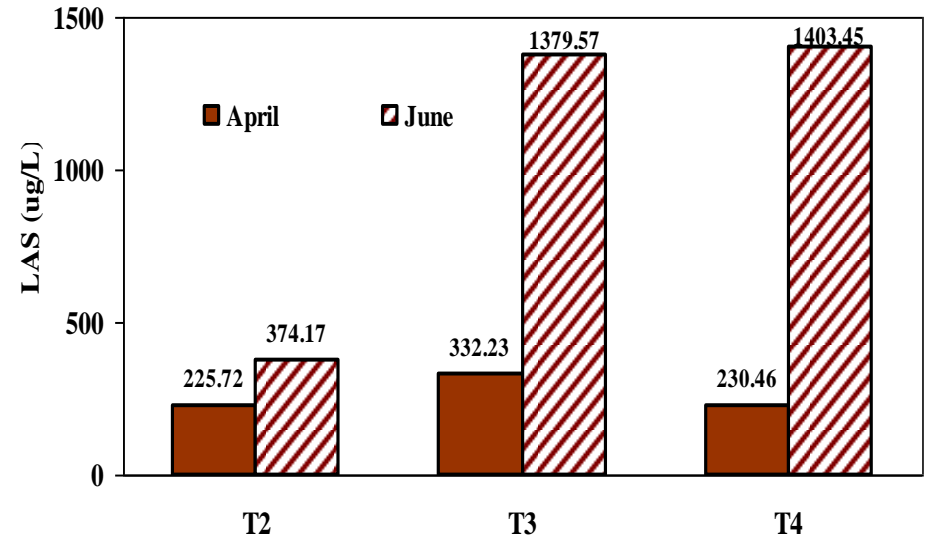


Figure A.14 LAS-Tall Fescue Graywater

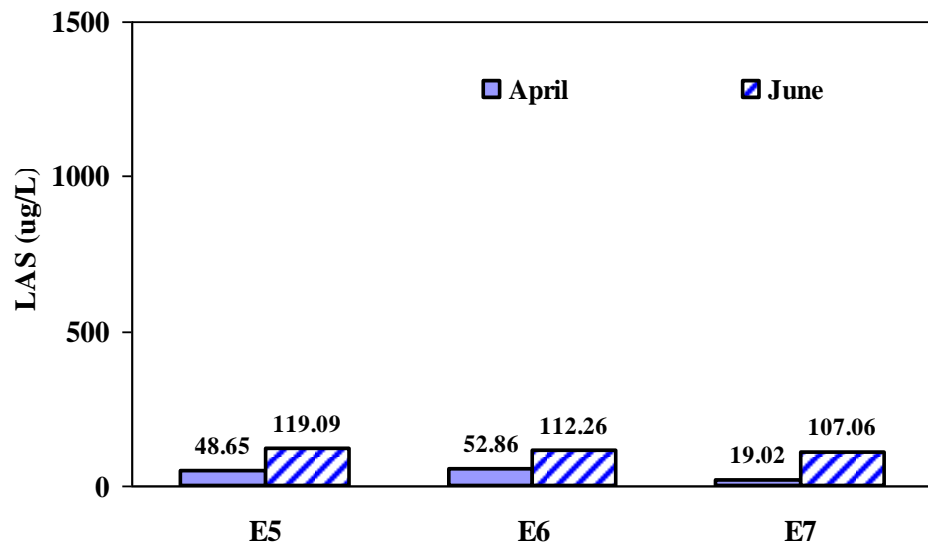


Figure A.15 LAS-Euonymous Potable Water

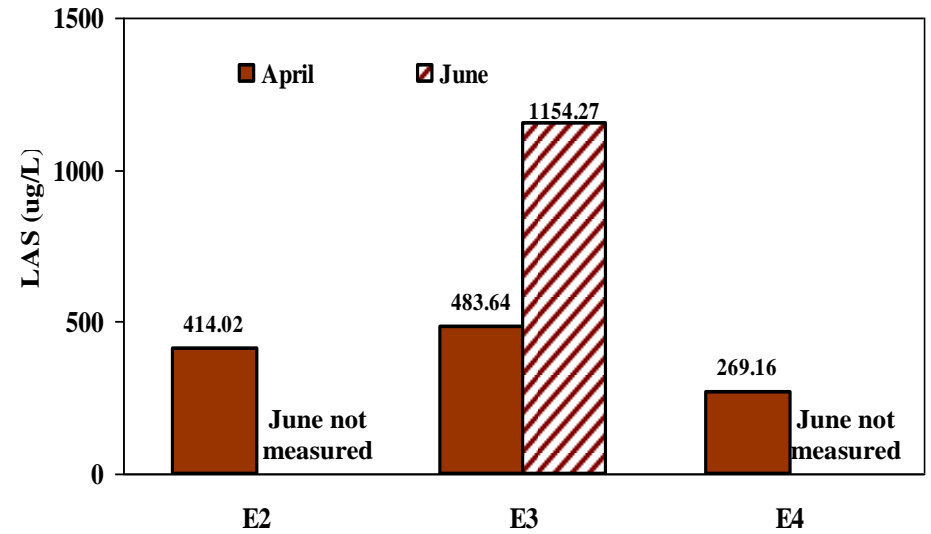


Figure A.16 LAS-Euonymous Graywater

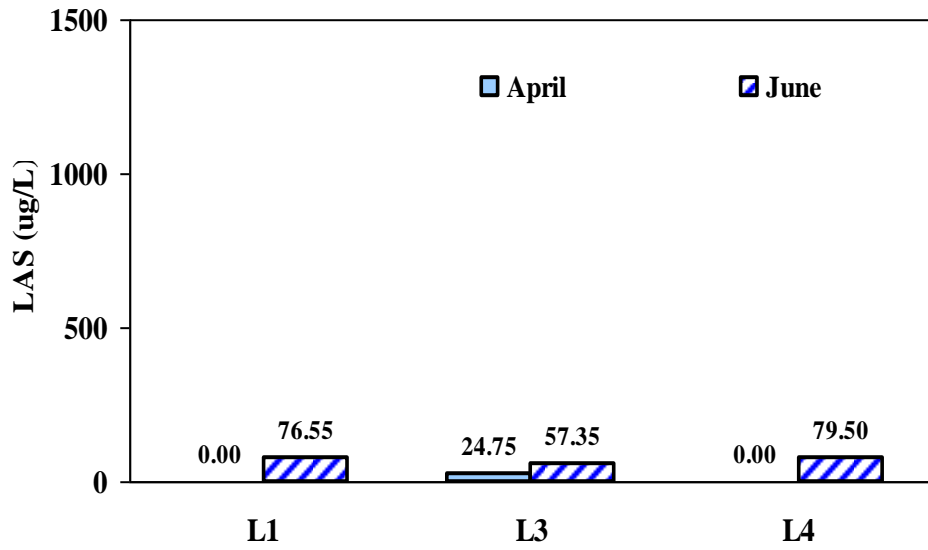


Figure A.17 LAS-Lemon Potable Water

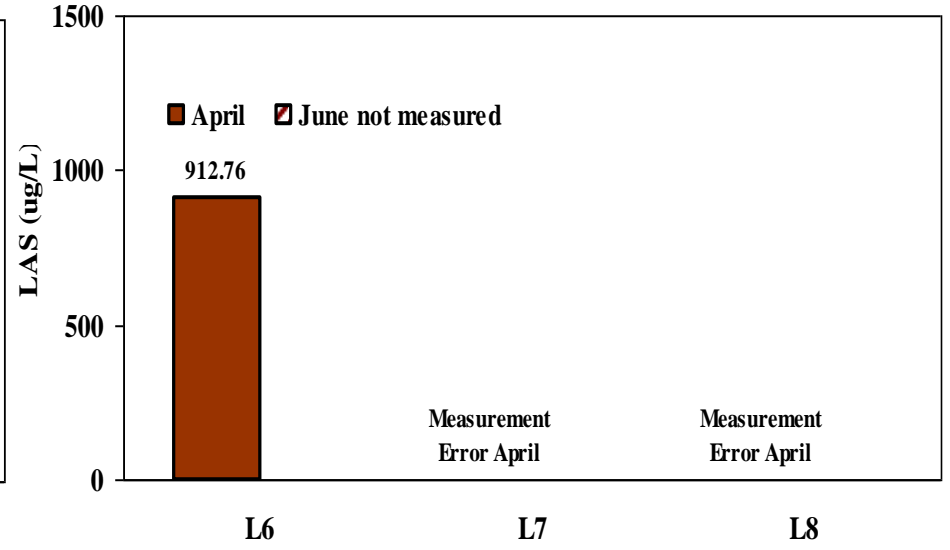


Figure A.18 LAS-Lemon Graywater

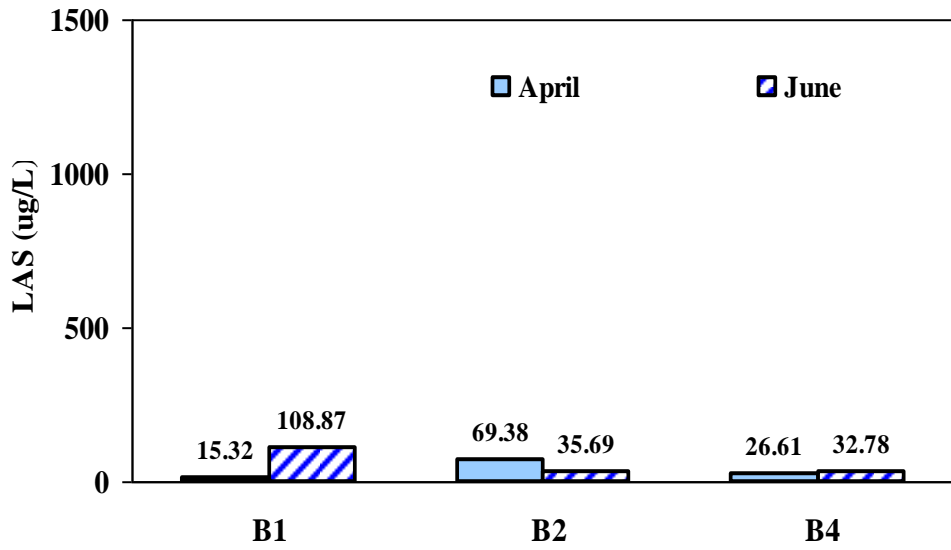


Figure A.19 LAS-Bermudagrass Potable Water

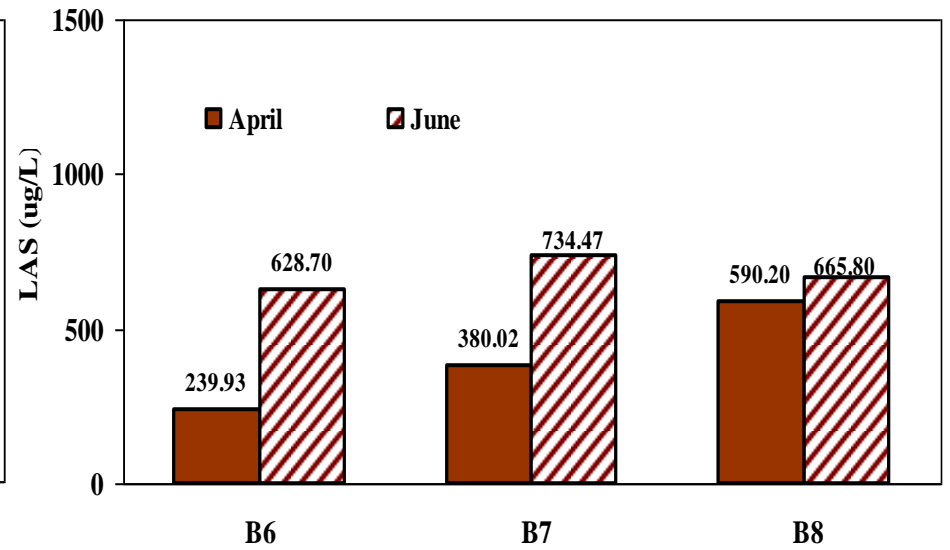


Figure A.20 LAS-Bermudagrass Graywater

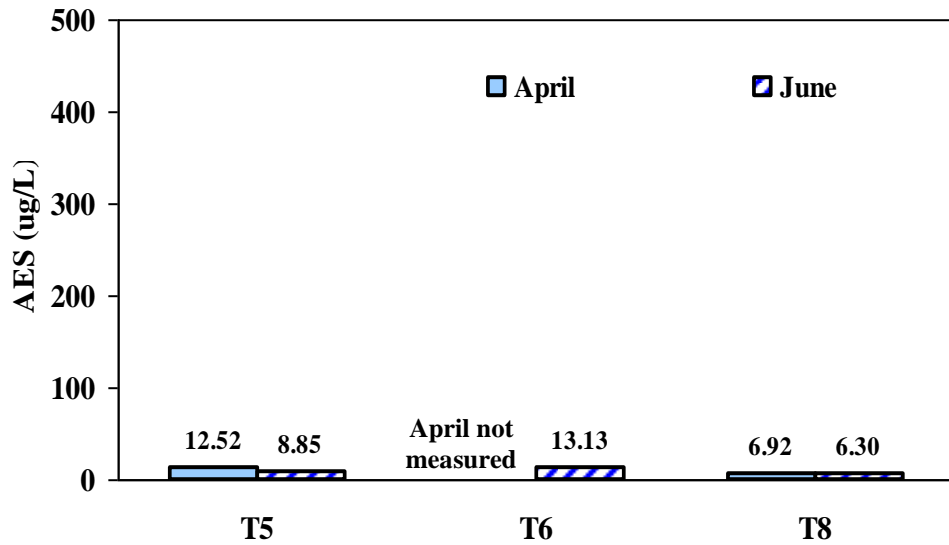


Figure A.21 AES-Tall Fescue Potable Water

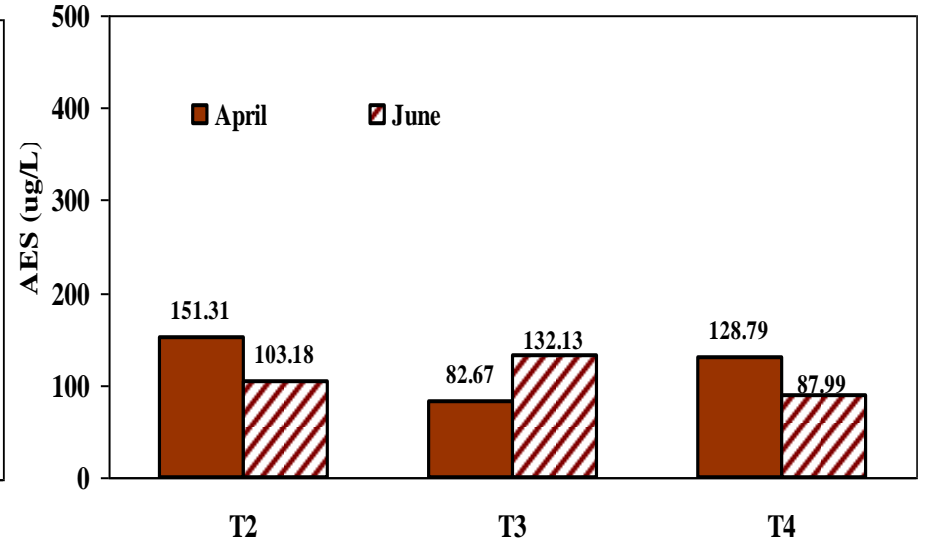


Figure A.22 AES-Tall Fescue Graywater

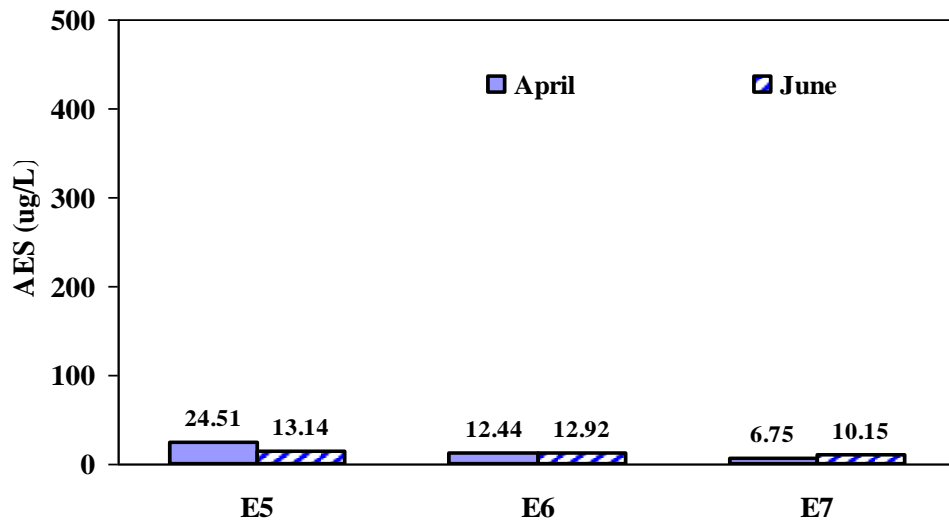


Figure A.23 AES-Euonymous Potable Water

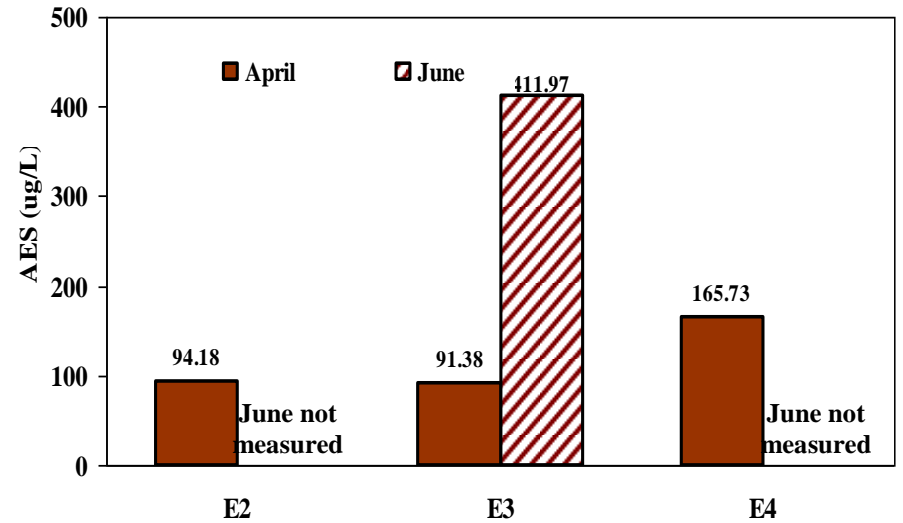


Figure A.24 AES-Euonymous Graywater

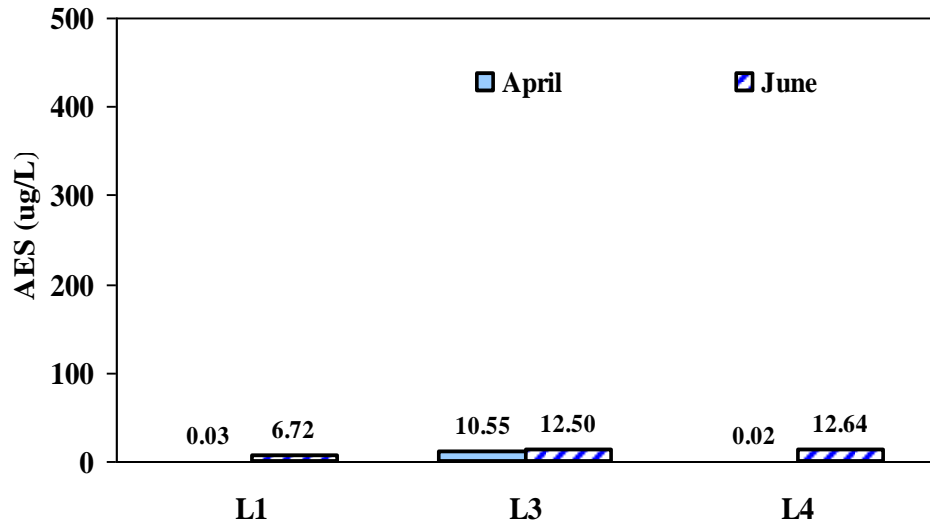


Figure A.25 AES-Lemon Potable Water

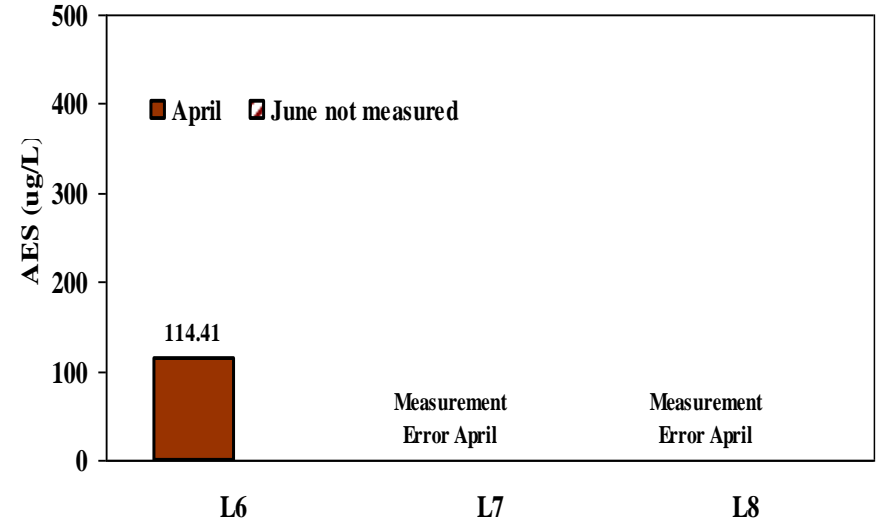


Figure A.26 AES-Lemon Graywater

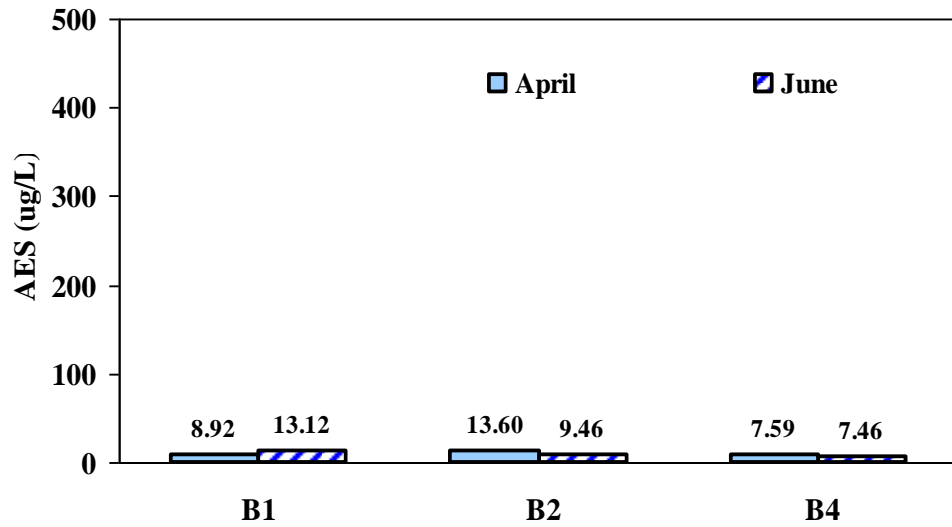


Figure A.27 AES-Bermudagrass Potable Water

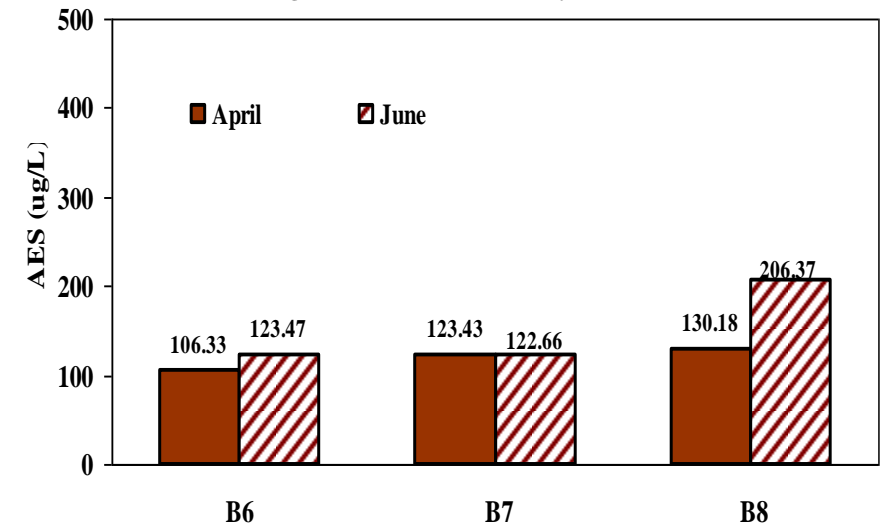


Figure A.28 AES-Bermudagrass Graywater

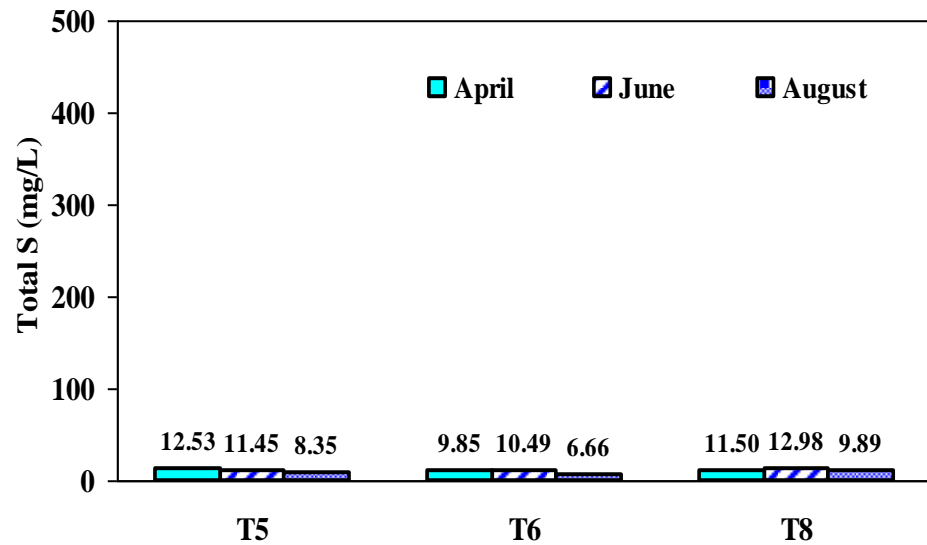


Figure A.29 Total S-Tall Fescue Potable Water

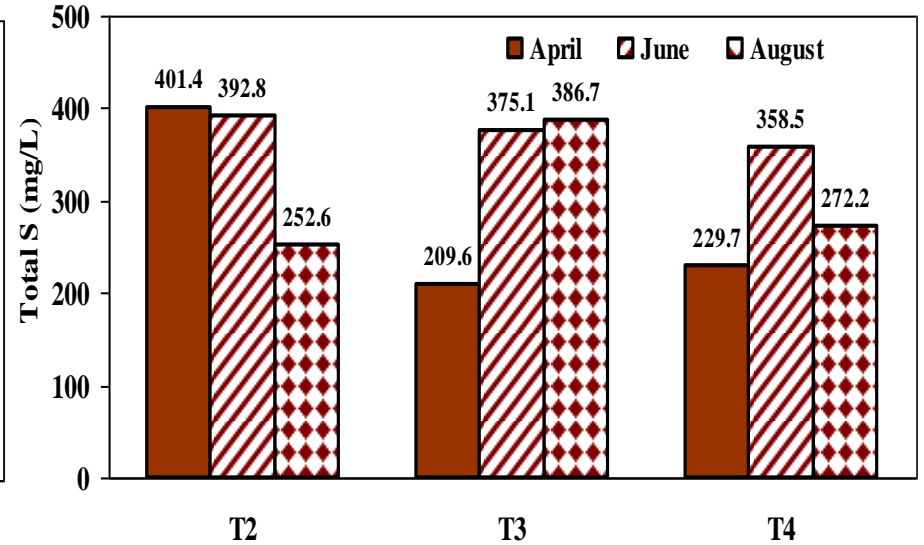


Figure A.30 Total S-Tall Fescue Graywater

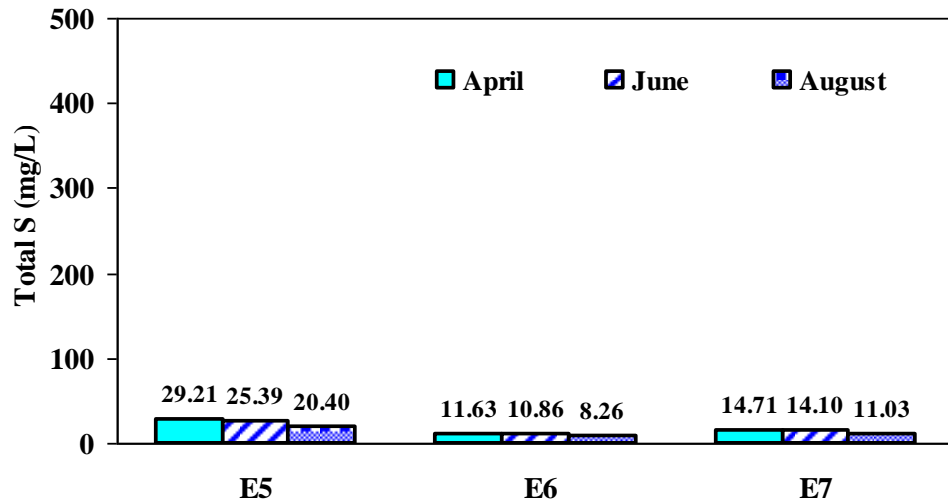


Figure A.31 Total S-Euonymous Potable Water

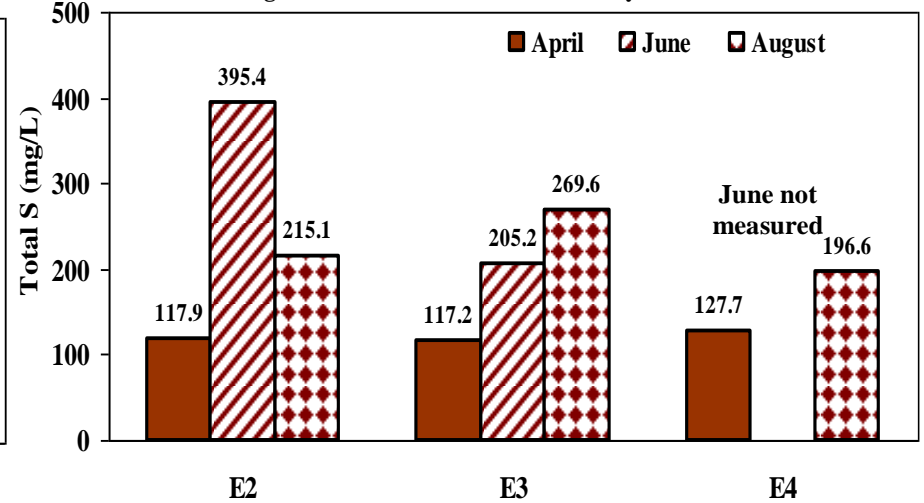


Figure A.32 Total S-Euonymous Graywater

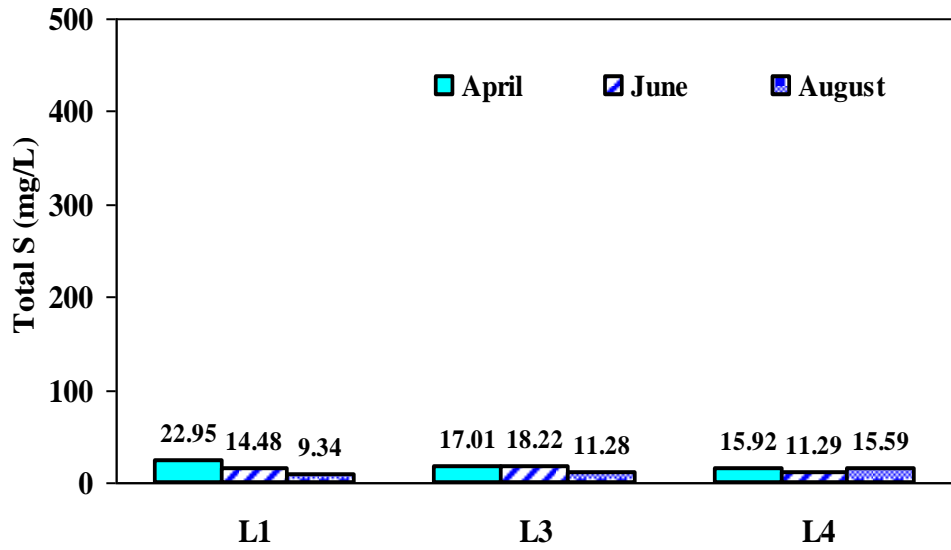


Figure A.33 Total S-Lemon Potable Water

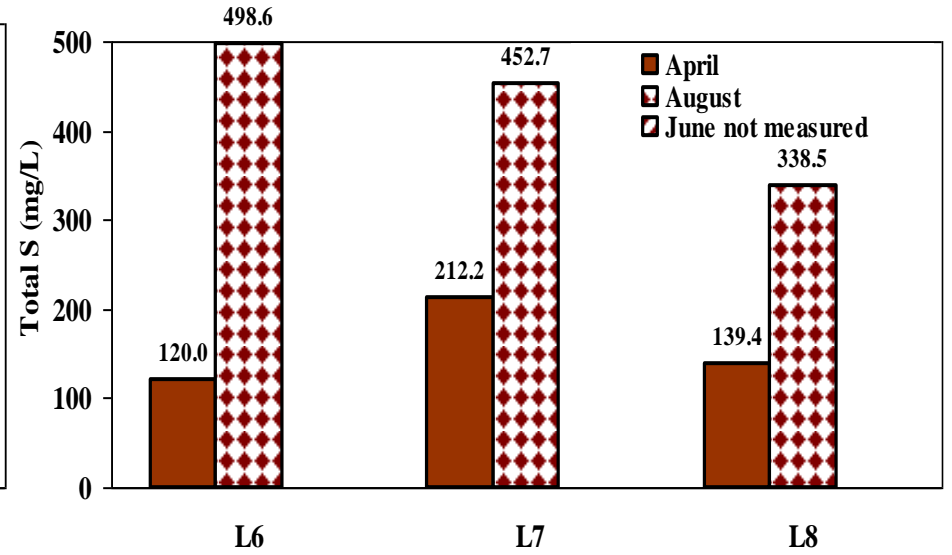


Figure A.34 Total S-Lemon Graywater

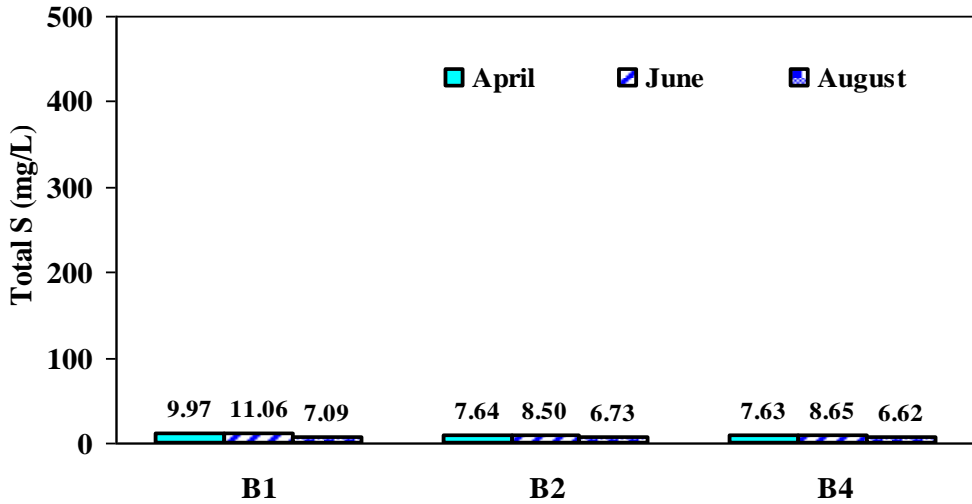


Figure A.35 Total S-Bermudagrass Potable Water

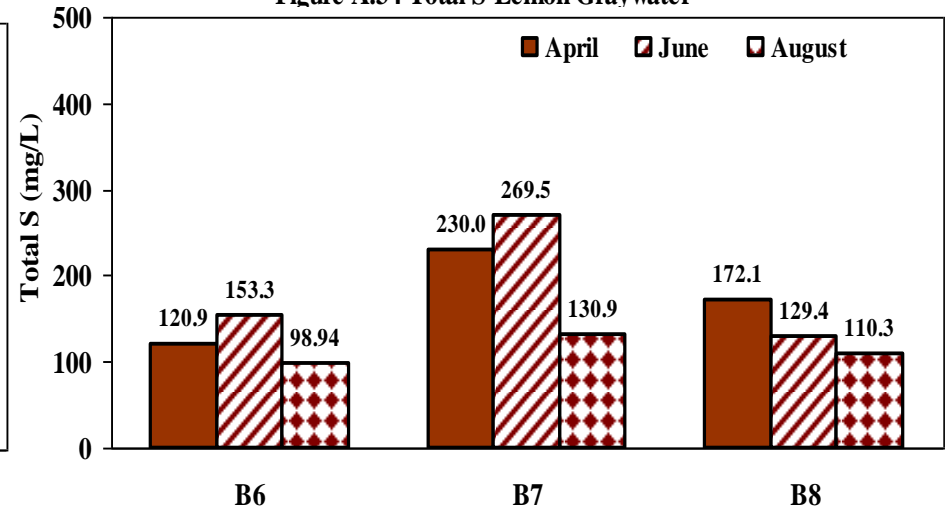


Figure A.36 Total S-Bermudagrass Graywater

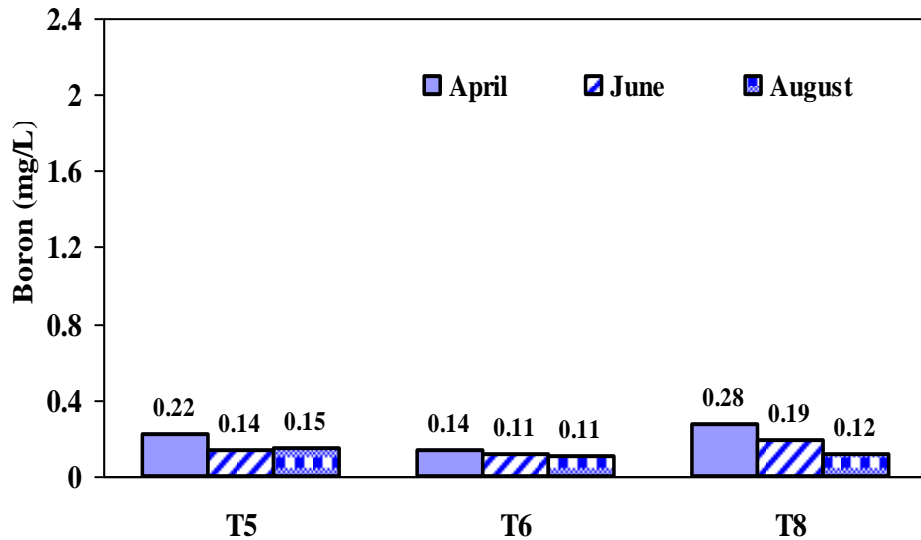


Figure A.37 Boron-Tall Fescue Potable Water

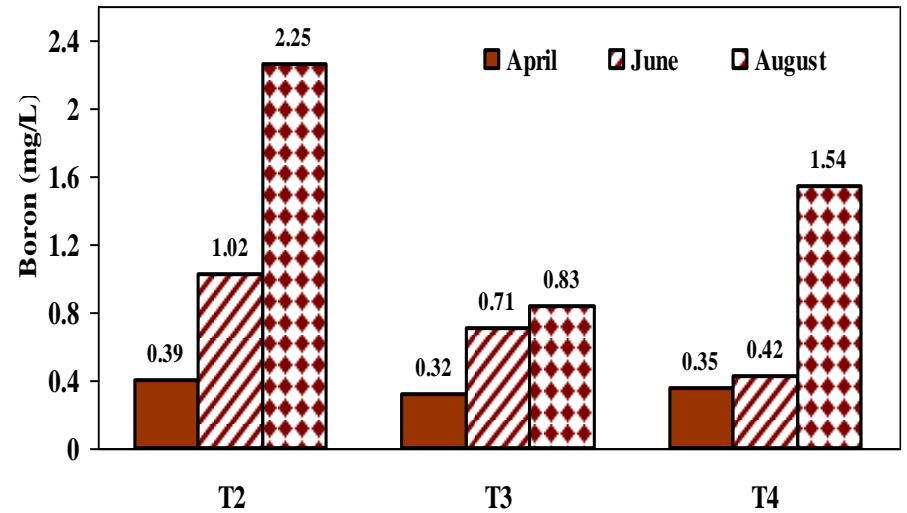


Figure A.38 Boron-Tall Fescue Graywater

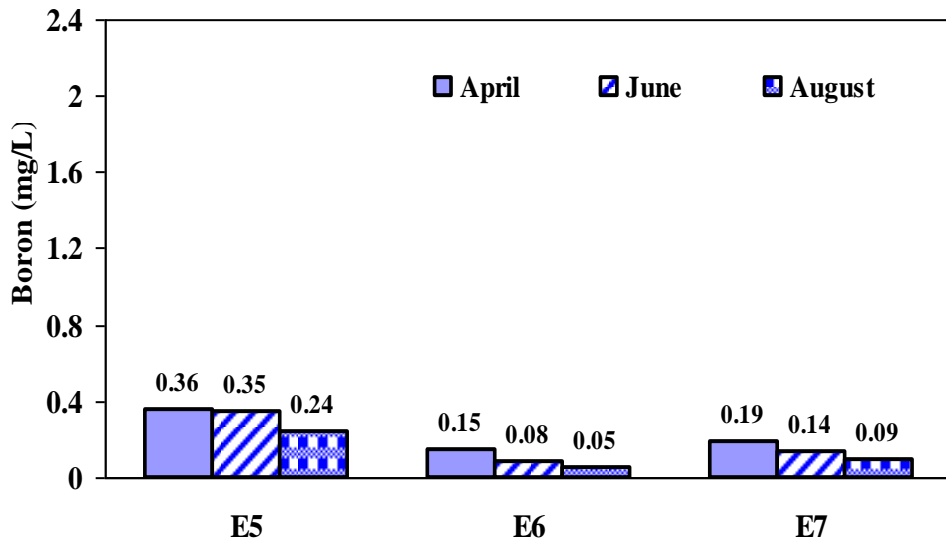


Figure A.39 Boron-Euonymous Potable Water

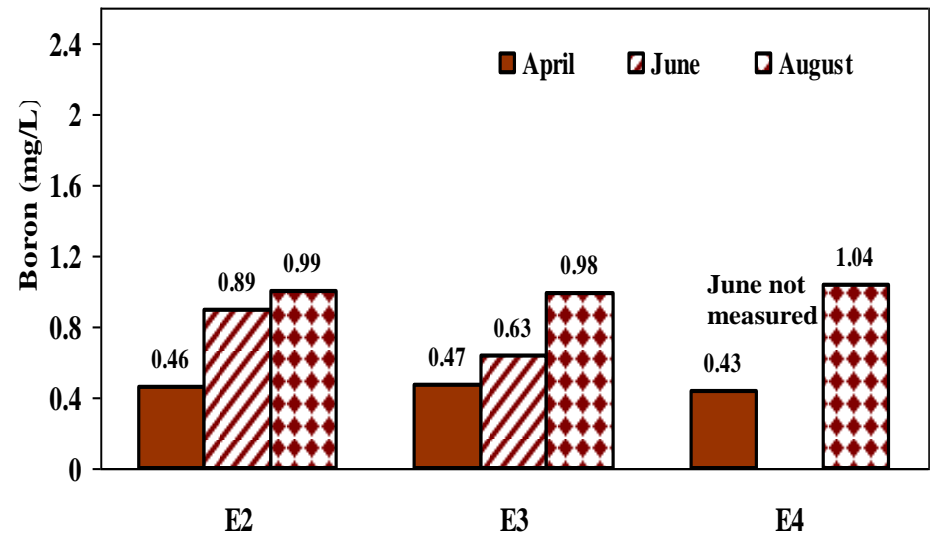


Figure A.40 Boron-Euonymous Graywater

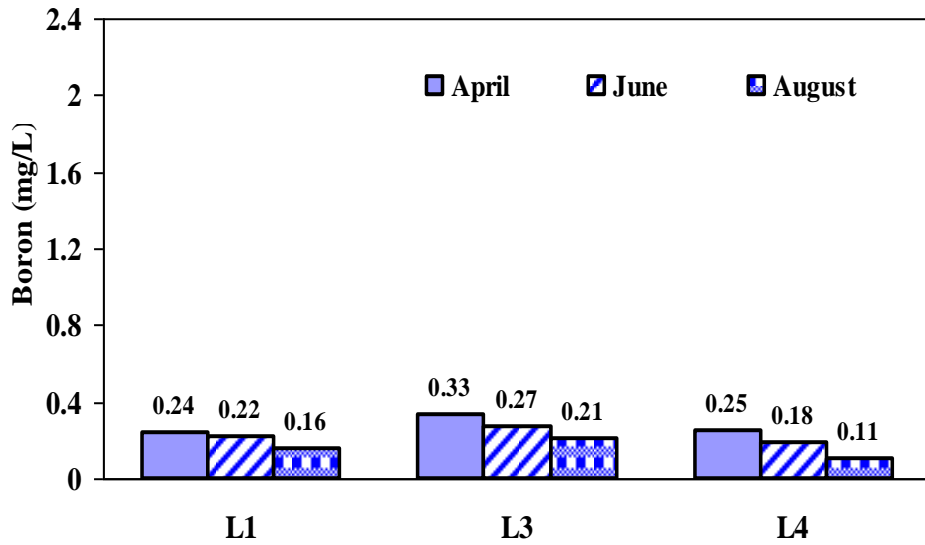


Figure A.41 Boron-Lemon Potable Water

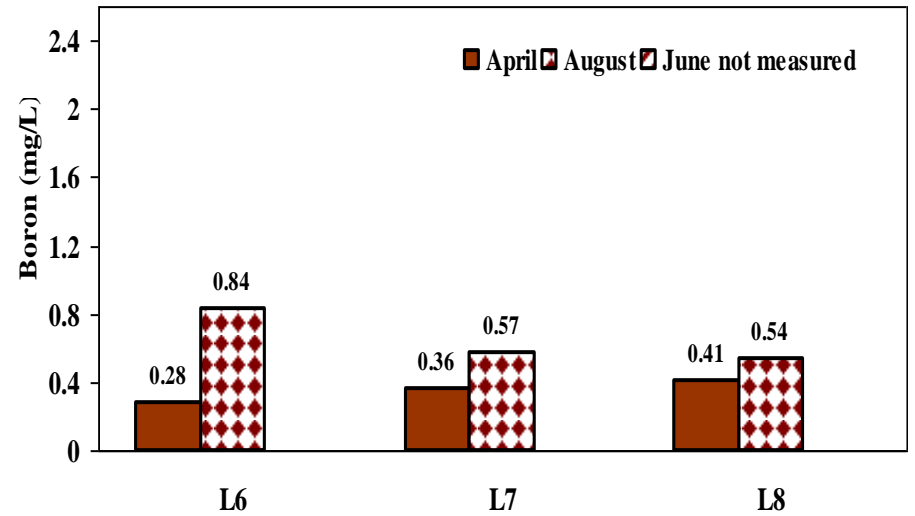


Figure A.42 Boron-Lemon Graywater

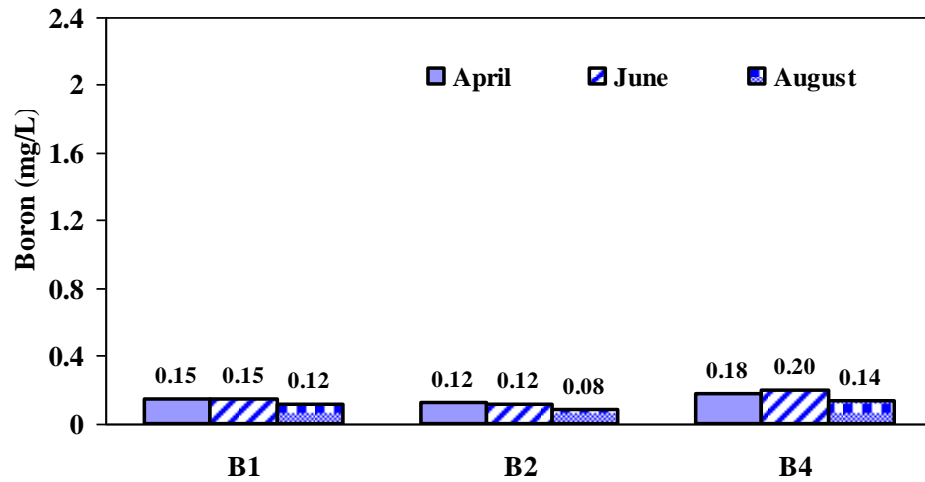


Figure A.43 Boron-Bermudagrass Potable Water

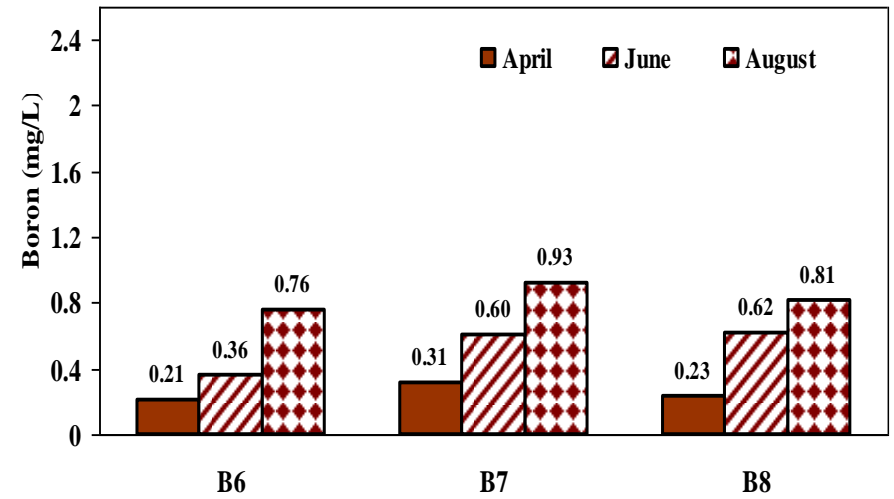


Figure A.44 Boron-Bermudagrass Graywater

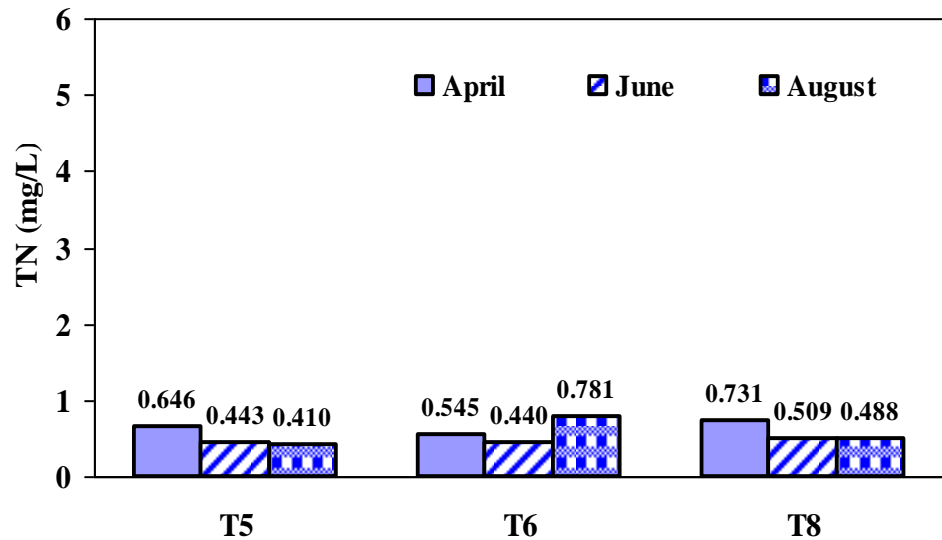


Figure A.45 TN-Tall Fescue Potable Water

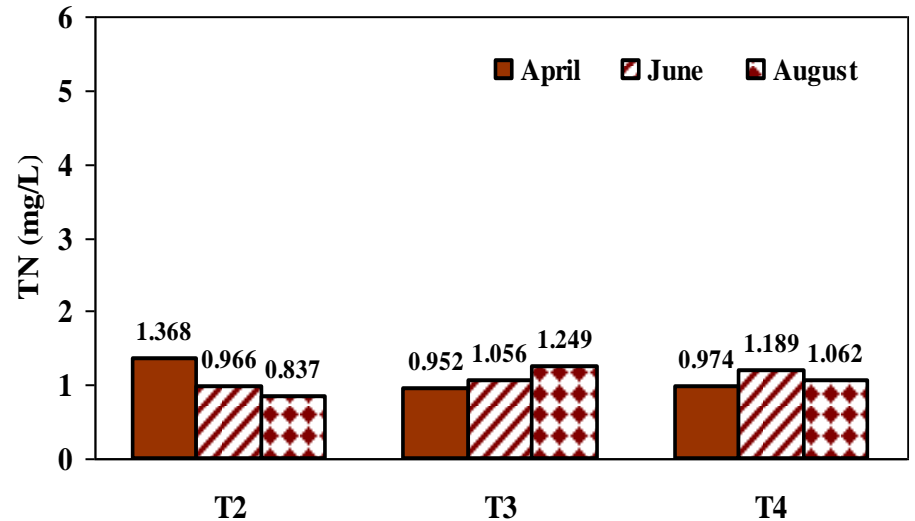


Figure A.46 TN-Tall Fescue Graywater

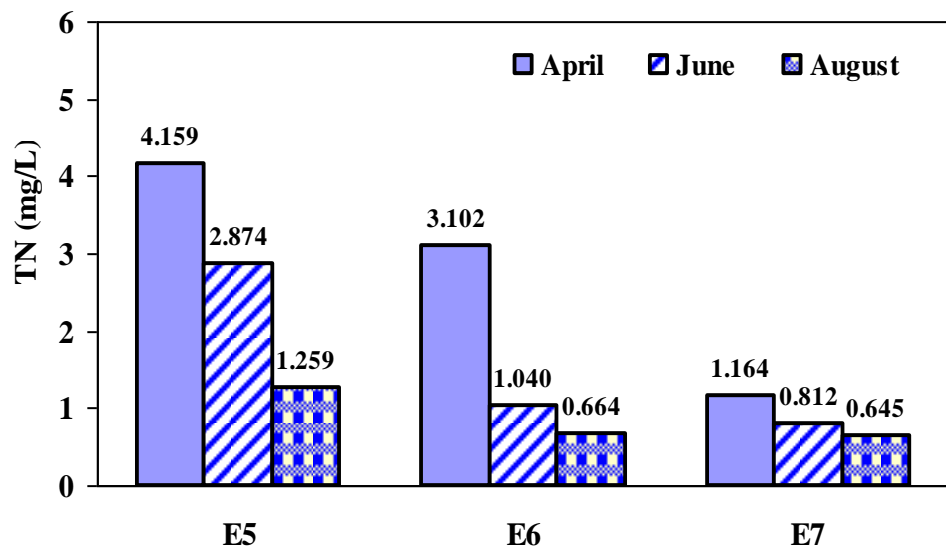


Figure A.47 TN- Euonymous Potable Water

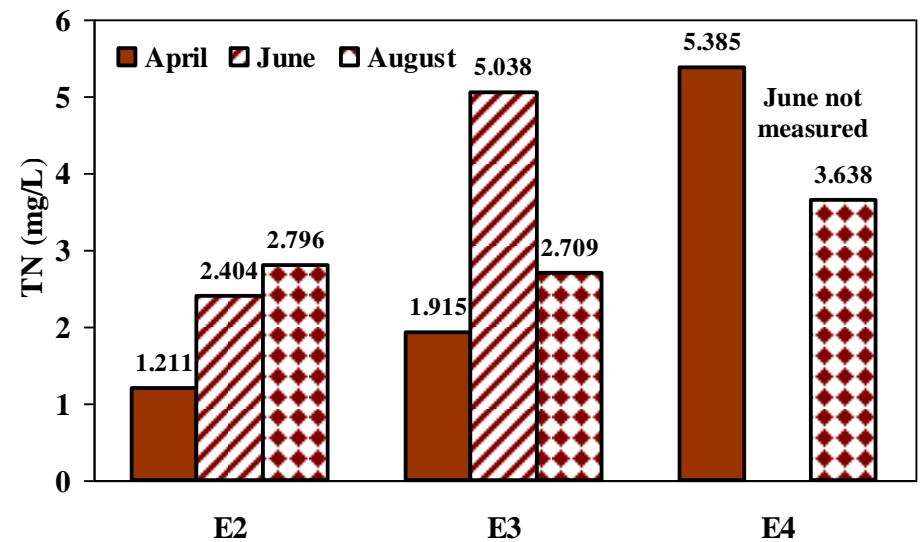


Figure A.48 TN- Euonymous Graywater

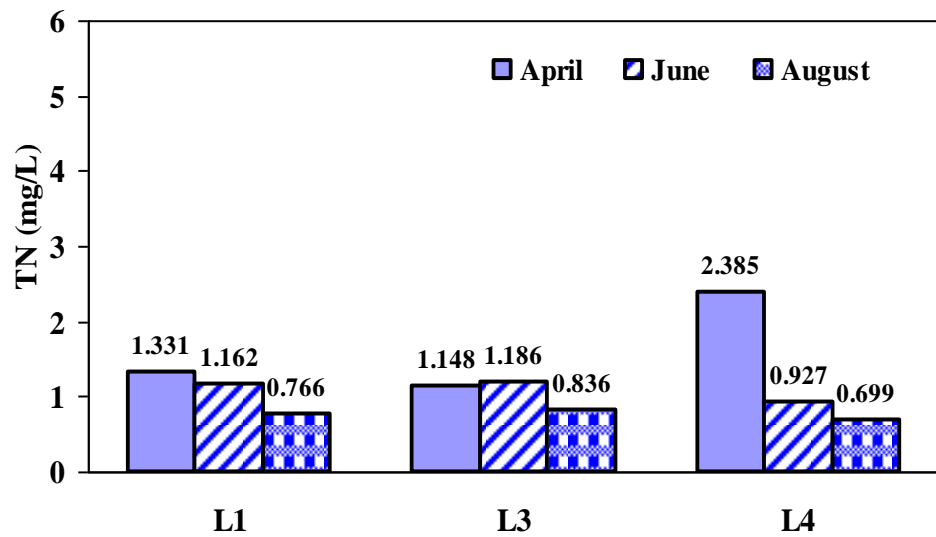


Figure A.49 TN- Lemon Potable Water

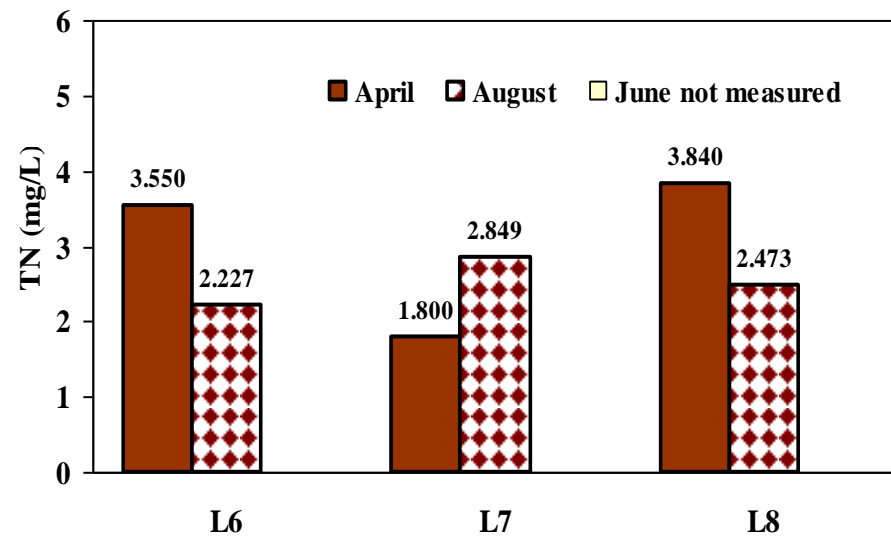


Figure A.50 TN- Lemon Graywater

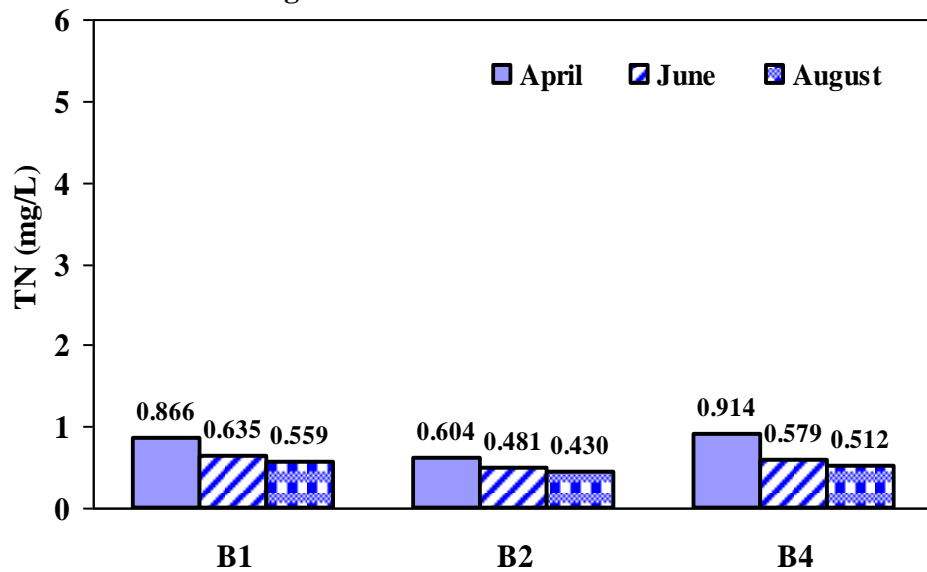


Figure A.51 TN- Bermudagrass Potable Water

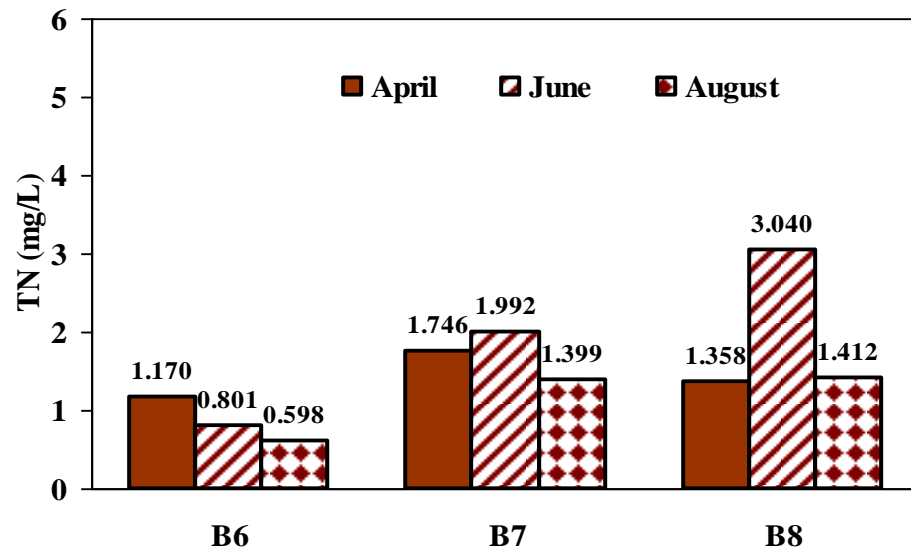


Figure A.52 TN- Bermudagrass Graywater

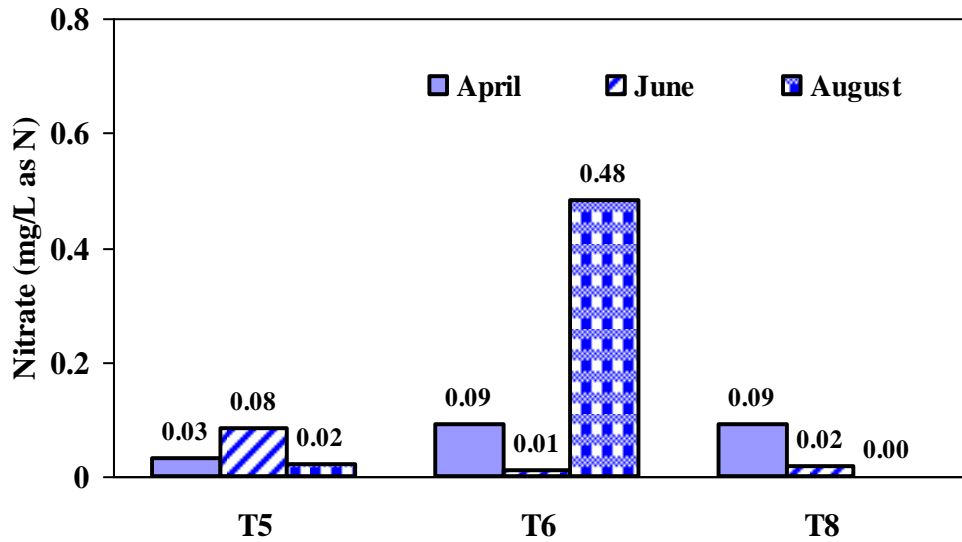


Figure A.53 Nitrate-Tall Fescue Potable Water

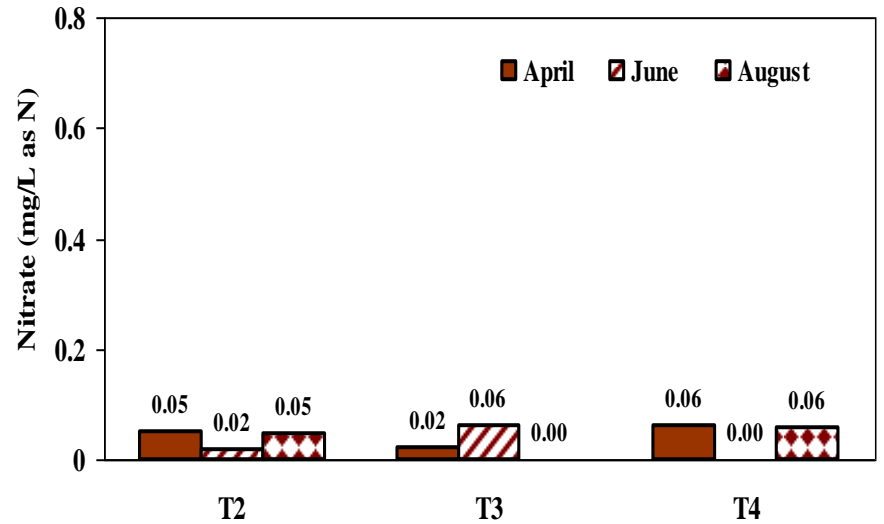


Figure A.54 Nitrate-Tall Fescue Graywater

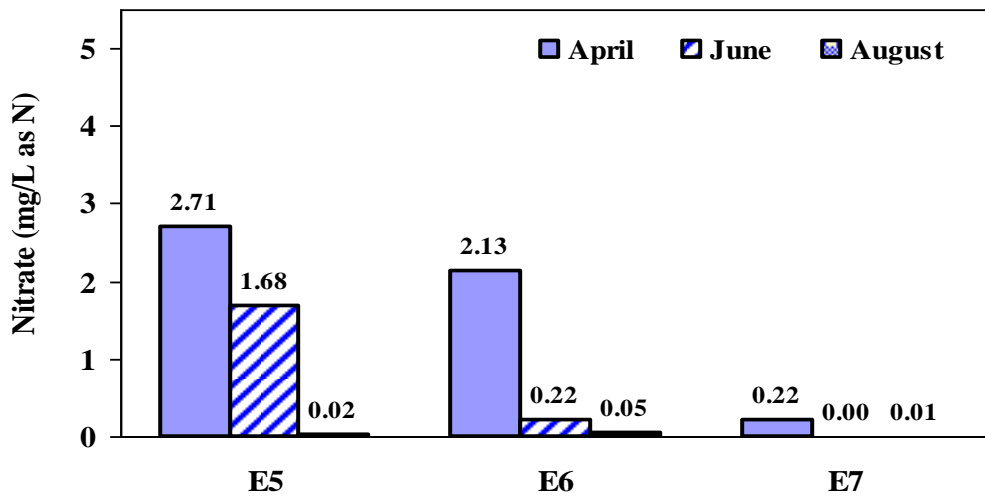


Figure A.55 Nitrate- Euonymous Potable Water

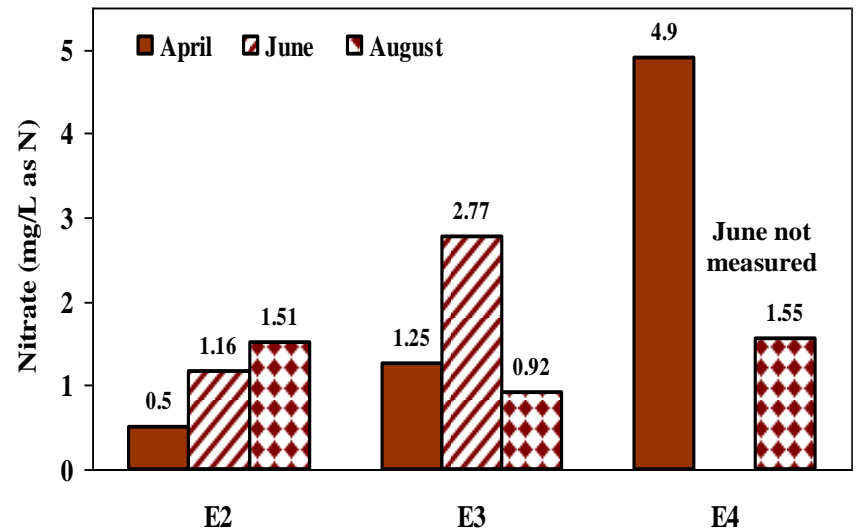


Figure A.56 Nitrate- Euonymous Graywater

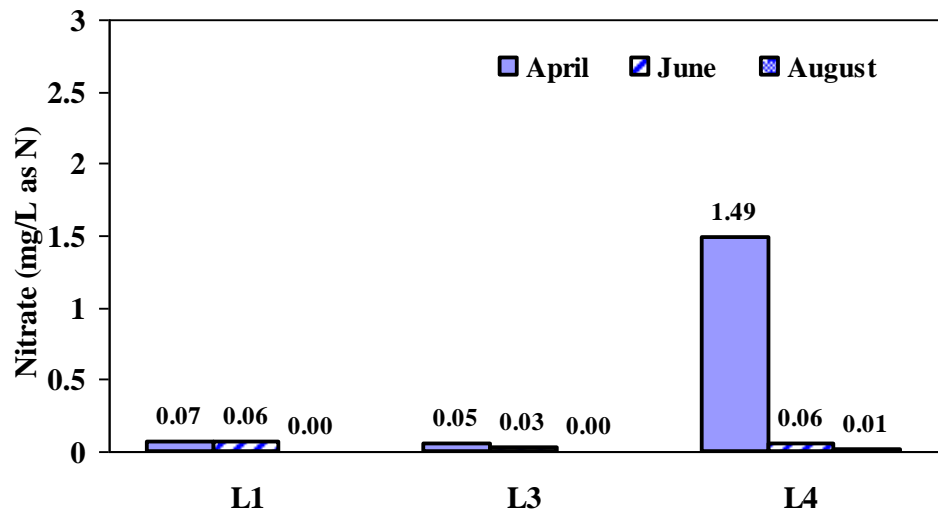


Figure A.57 Nitrate- Lemon Potable Water

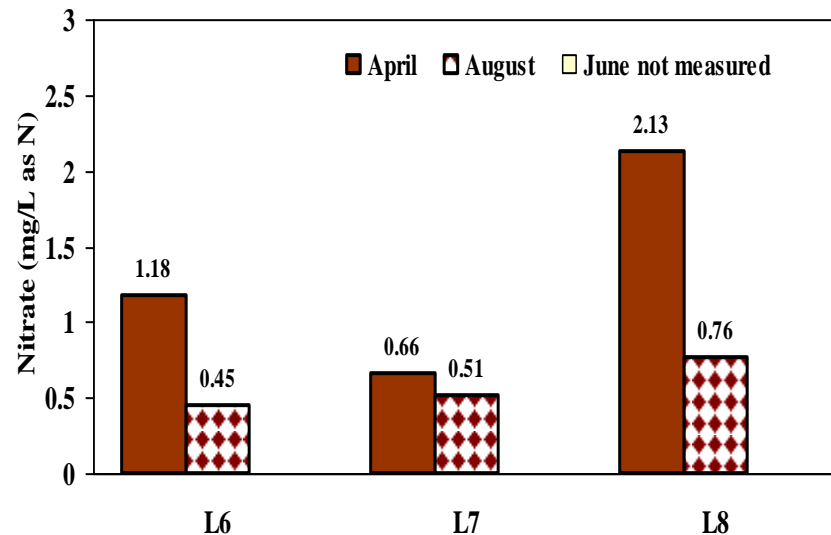


Figure A.58 Nitrate- Lemon Graywater

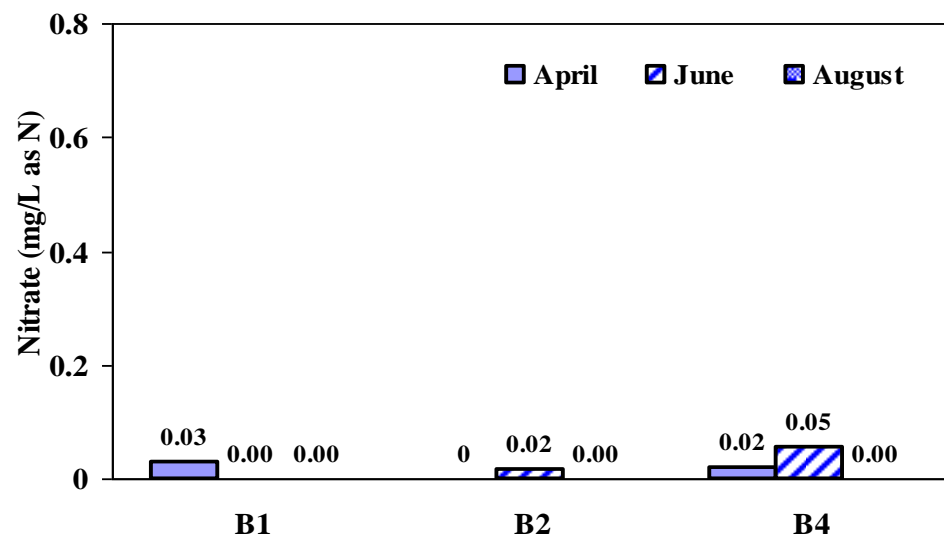


Figure A.59 Nitrate- Bermudagrass Potable Water

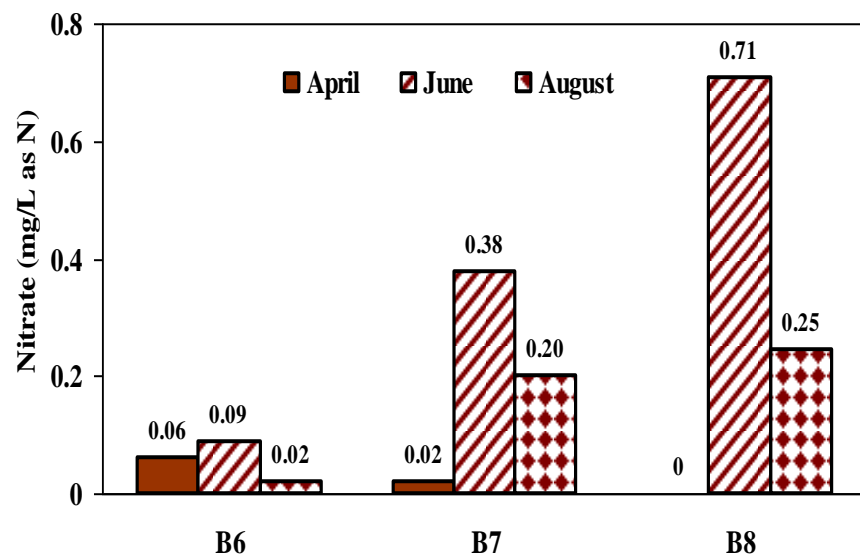


Figure A.60 Nitrate- Bermudagrass Graywater

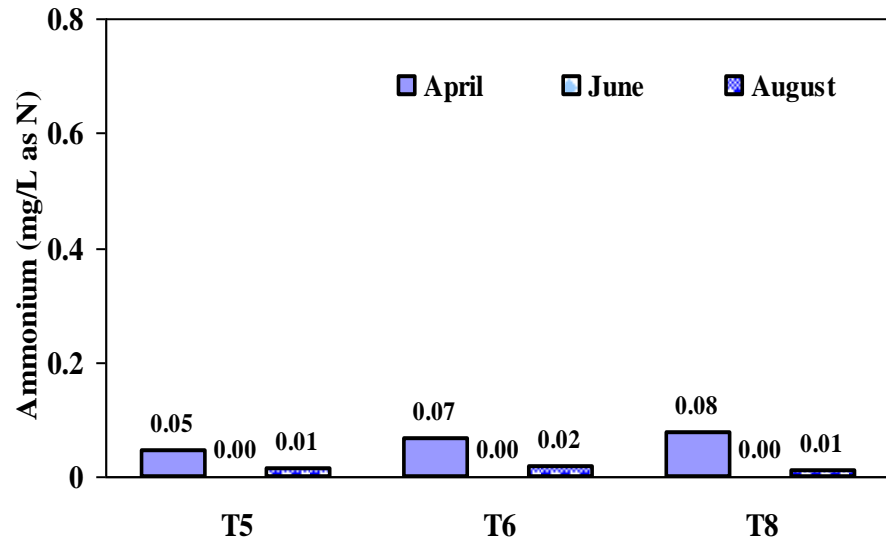


Figure A.61 Ammonium-Tall Fescue Potable Water

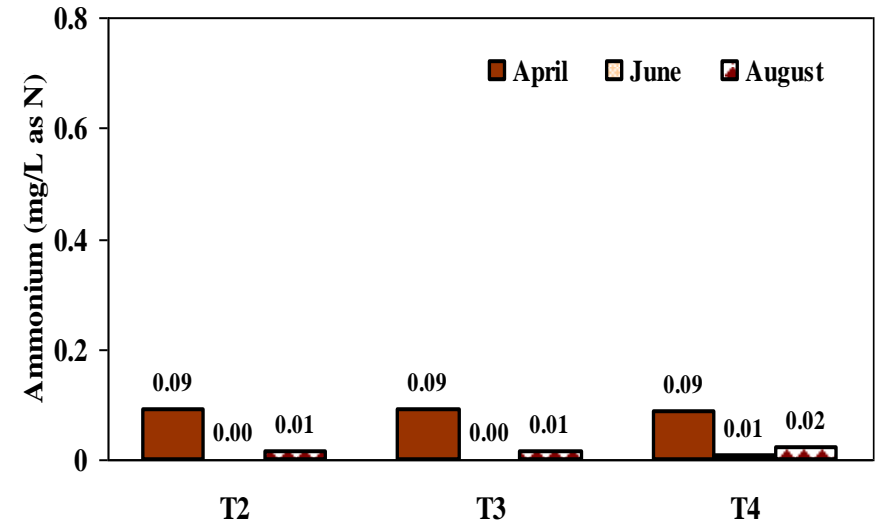


Figure A.62 Ammonium-Tall Fescue Graywater

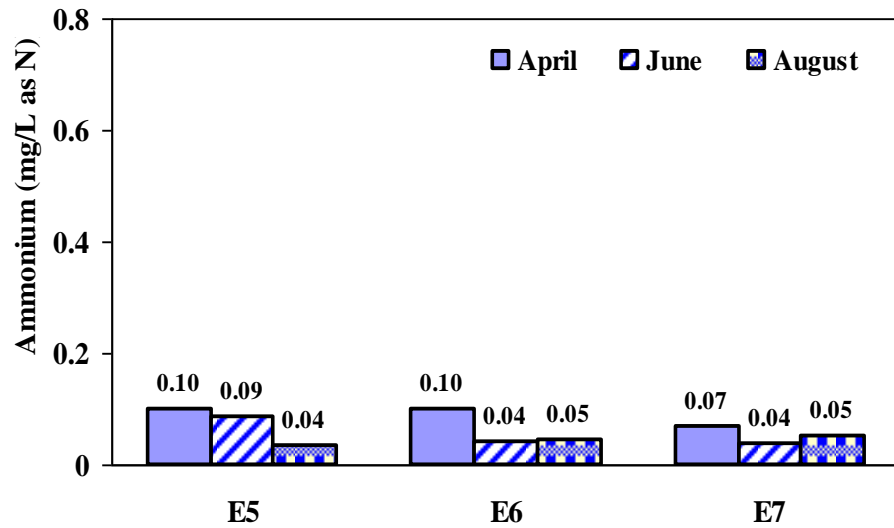


Figure A.63 Ammonium- Euonymous Potable Water

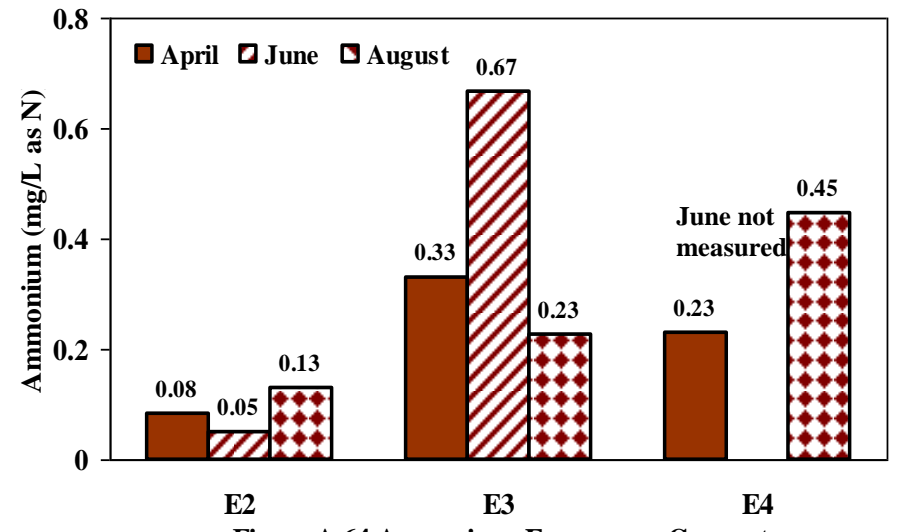


Figure A.64 Ammonium- Euonymous Graywater

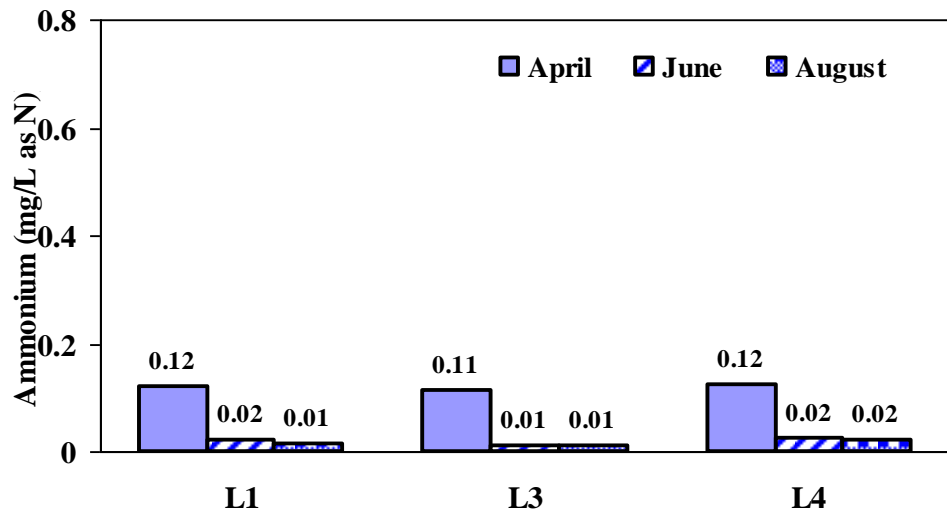


Figure A.65 Ammonium- Lemon Potable Water

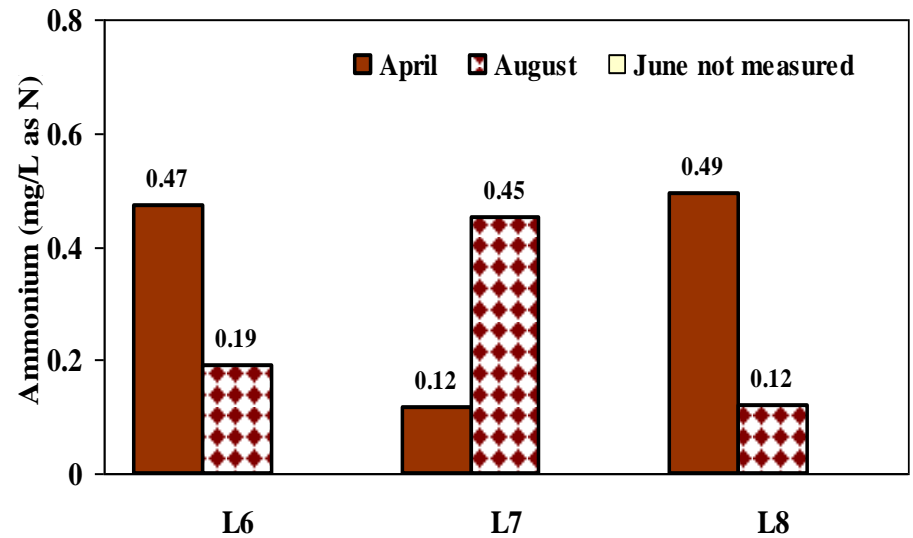


Figure A.66 Ammonium- Lemon Graywater

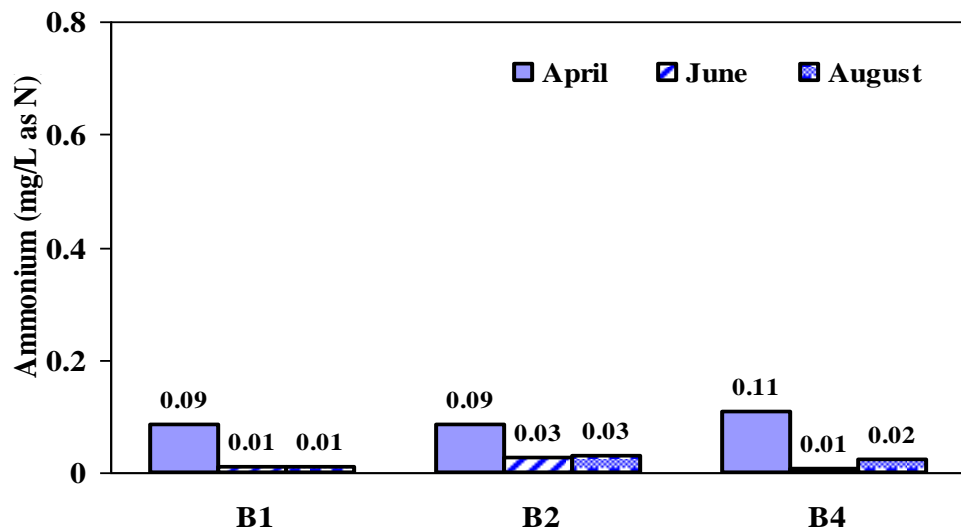


Figure A.67 Ammonium- Bermudagrass Potable Water

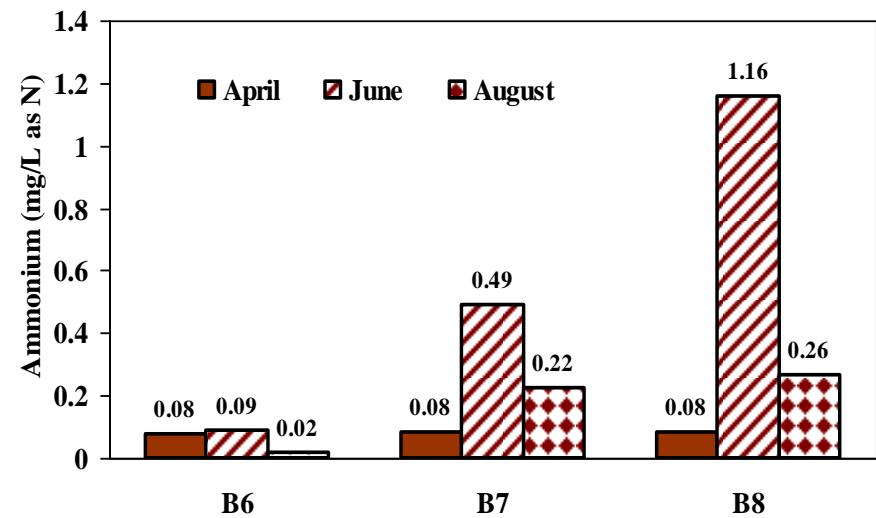


Figure A.68 Ammonium- Bermudagrass Graywater

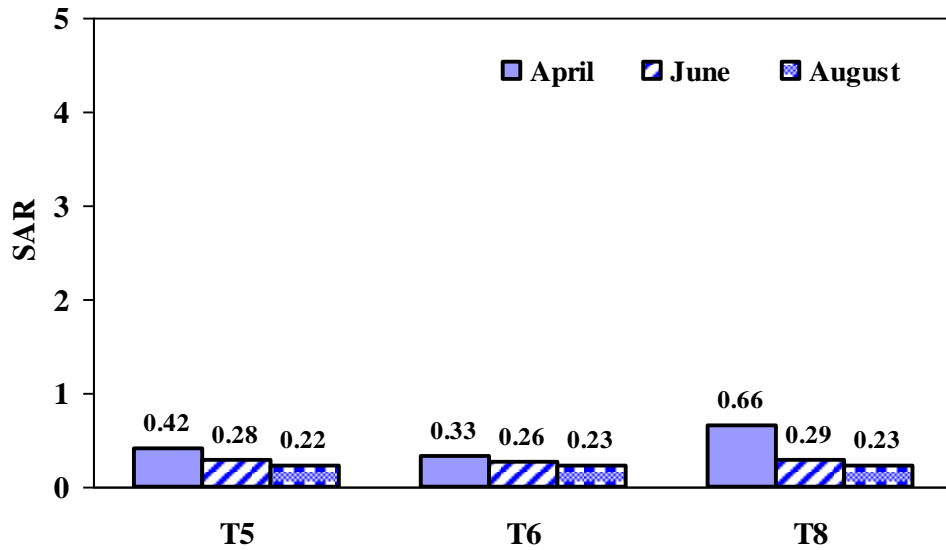


Figure A.69 SAR- Tall Fescue Potable Water

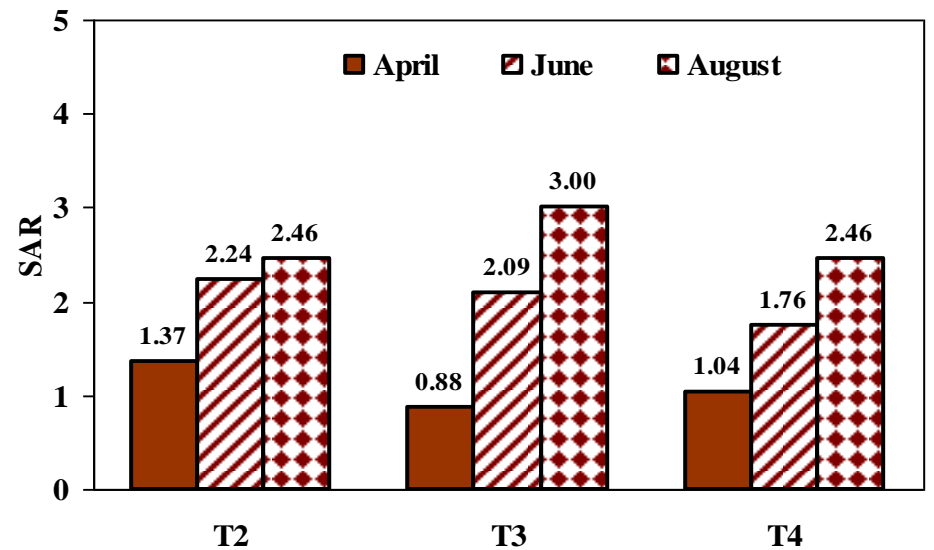


Figure A.70 SAR- Tall Fescue Graywater

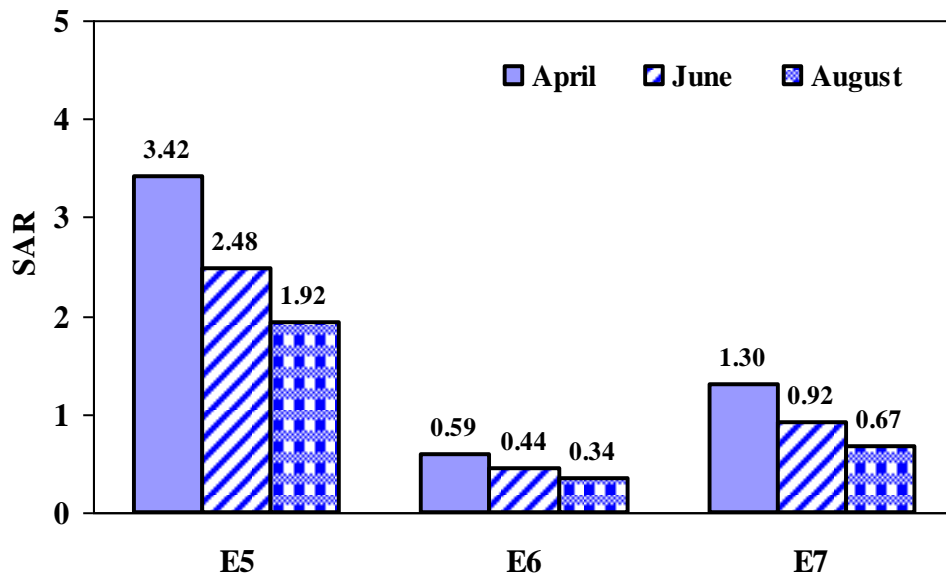


Figure A.71 SAR- Euonymous Potable Water

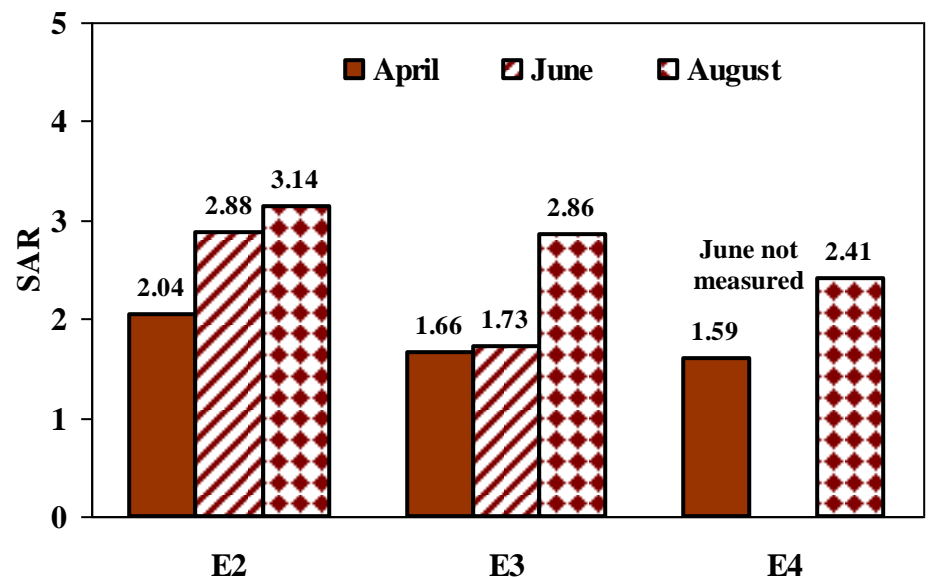


Figure A.72 SAR- Euonymous Graywater

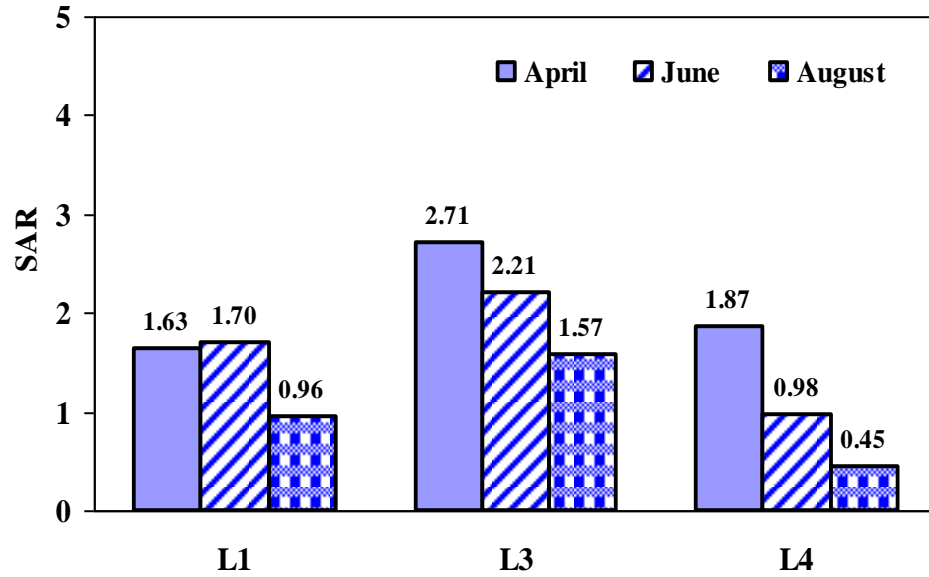


Figure A.73 SAR- Lemon Potable Water

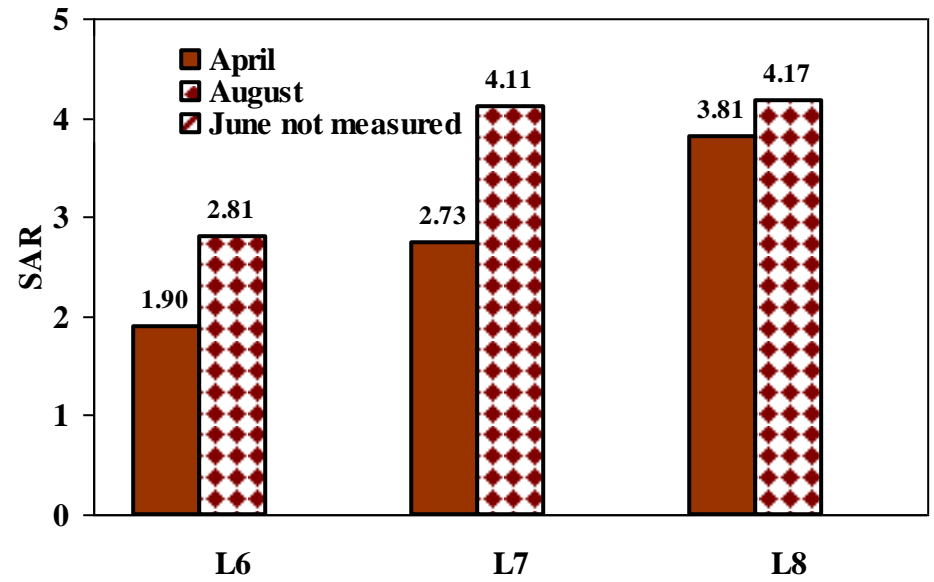


Figure A.74 SAR- Lemon Graywater

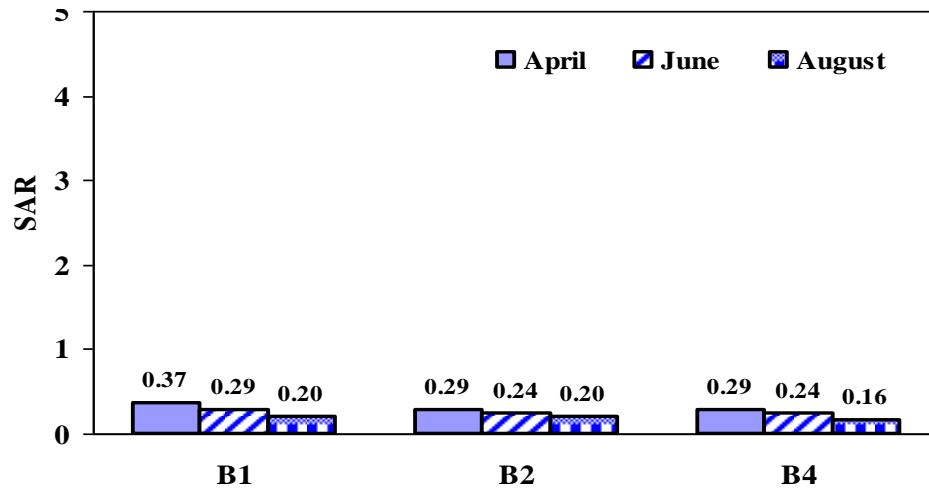


Figure A.75 SAR- Bermudagrass Potable Water

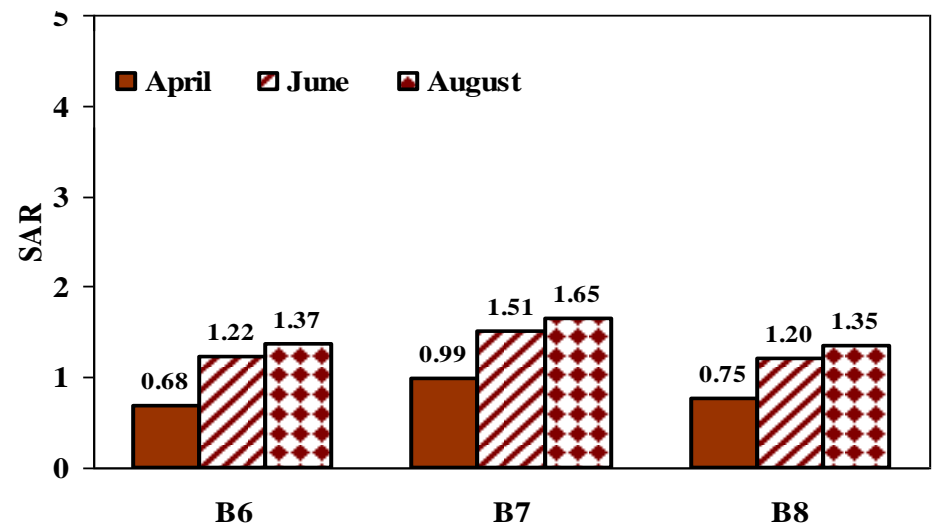


Figure A.76 SAR- Bermudagrass Graywater

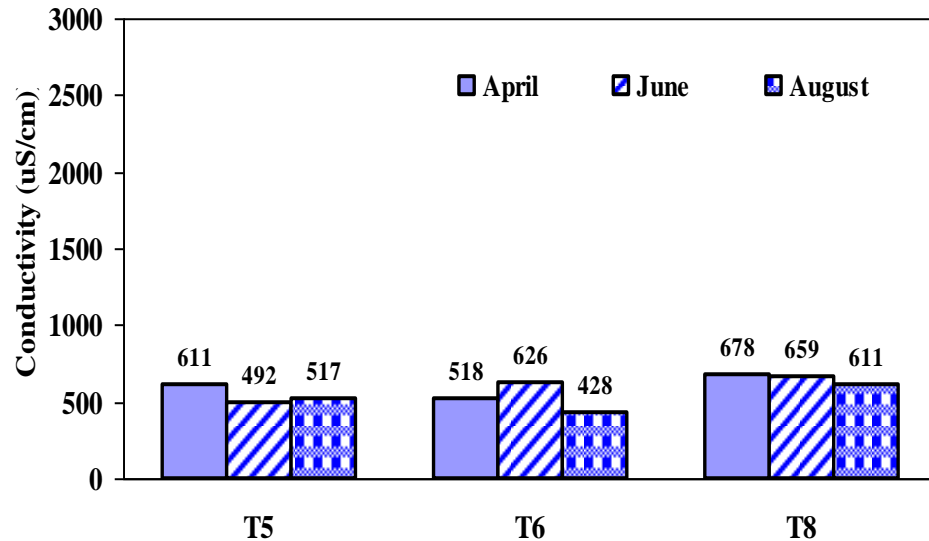


Figure A.77 Conductivity-Tall Fescue Potable Water

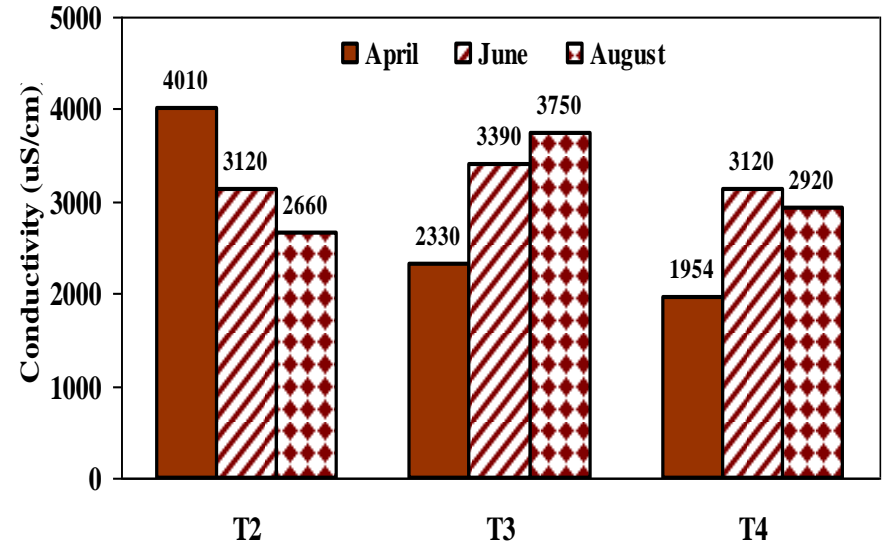


Figure A.78 Conductivity-Tall Fescue Graywater

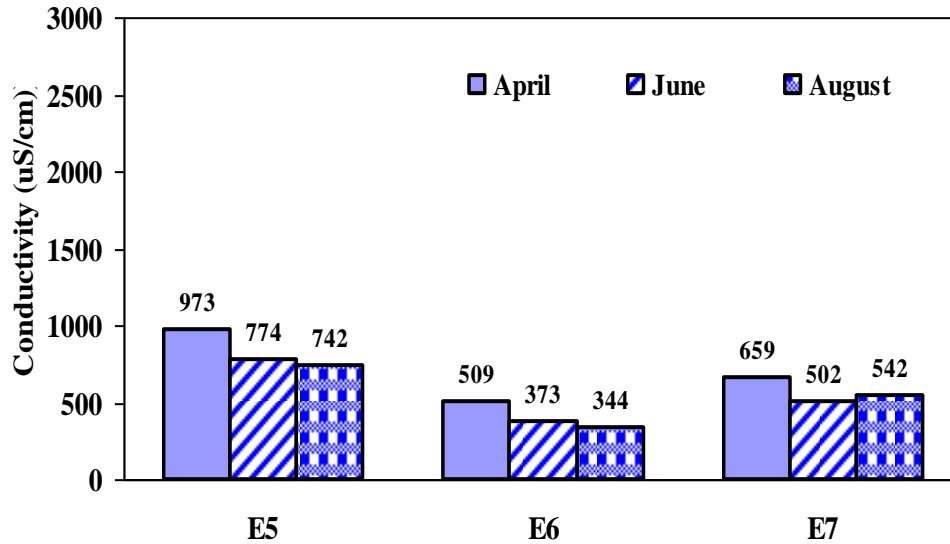


Figure A.79 Conductivity-Euonymous Potable Water

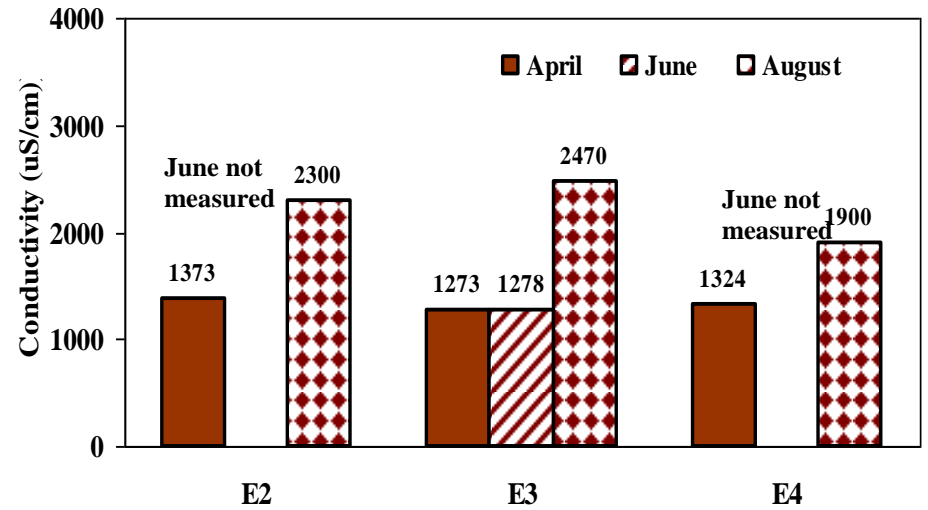


Figure A.80 Conductivity-Euonymous Graywater

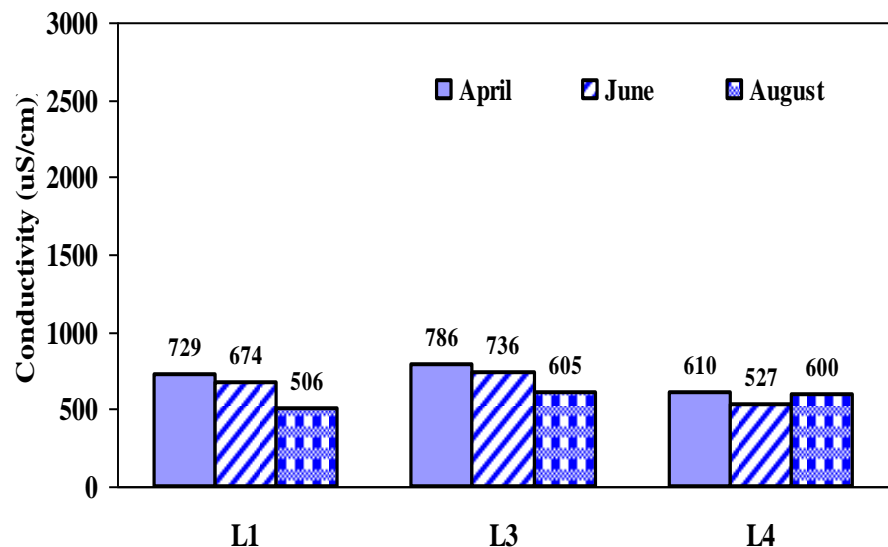


Figure A.81 Conductivity-Lemon Potable Water

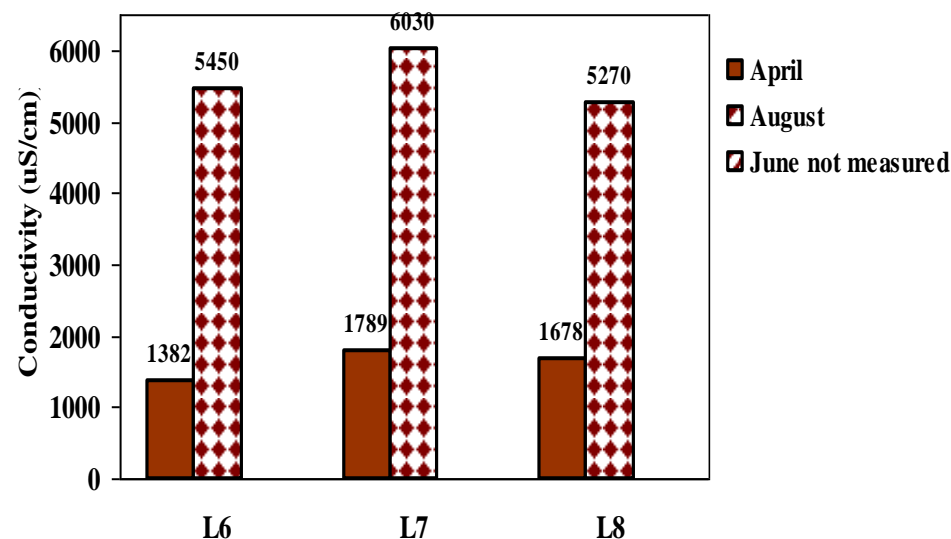


Figure A.82 Conductivity-Lemon Graywater

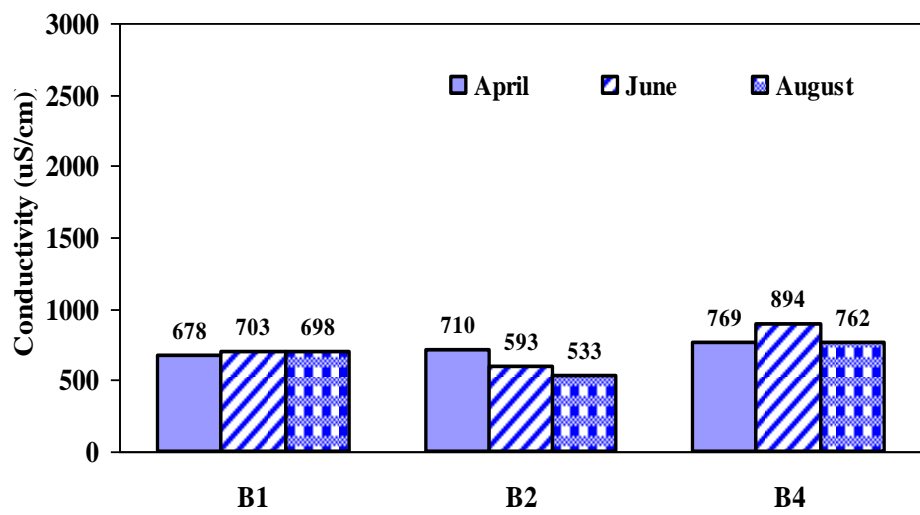


Figure A.83 Conductivity-Bermudagrass Potable Water

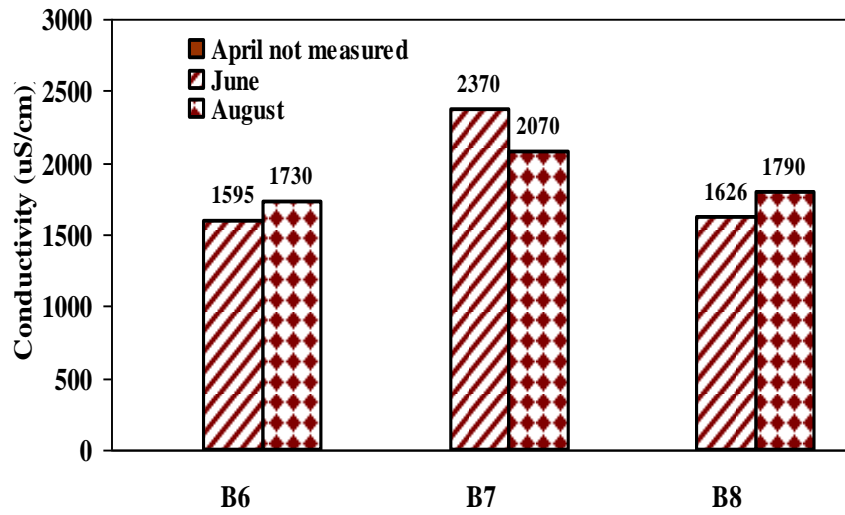


Figure A.84 Conductivity-Bermudagrass Graywater

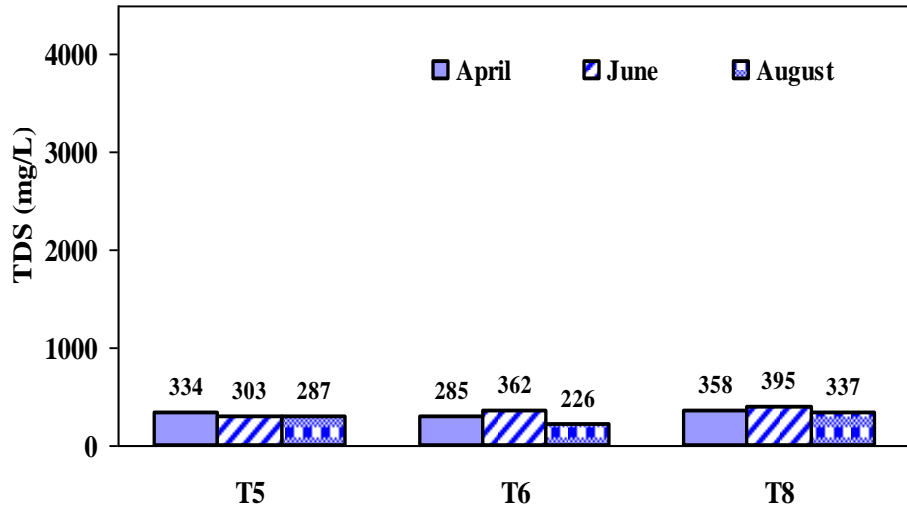


Figure A.85 TDS-Tall Fescue Potable Water

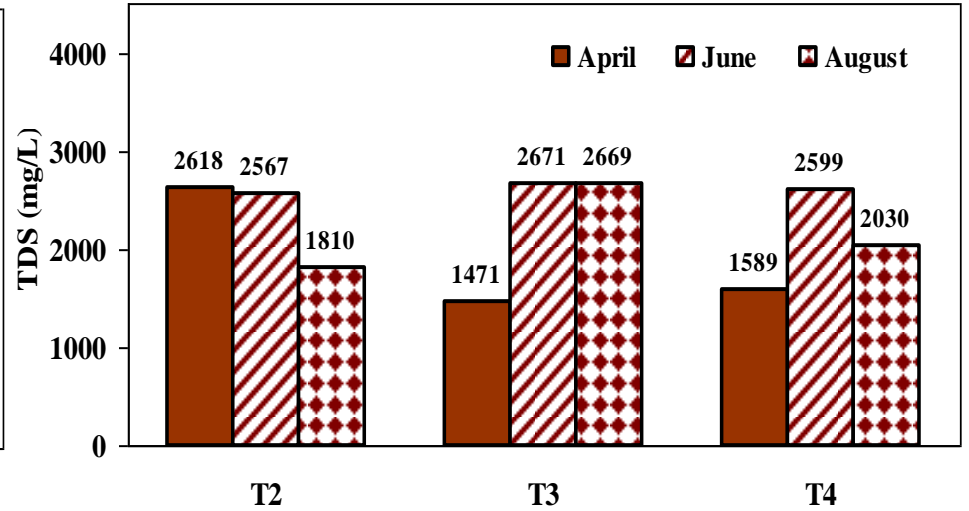


Figure A.86 TDS-Tall Fescue Graywater

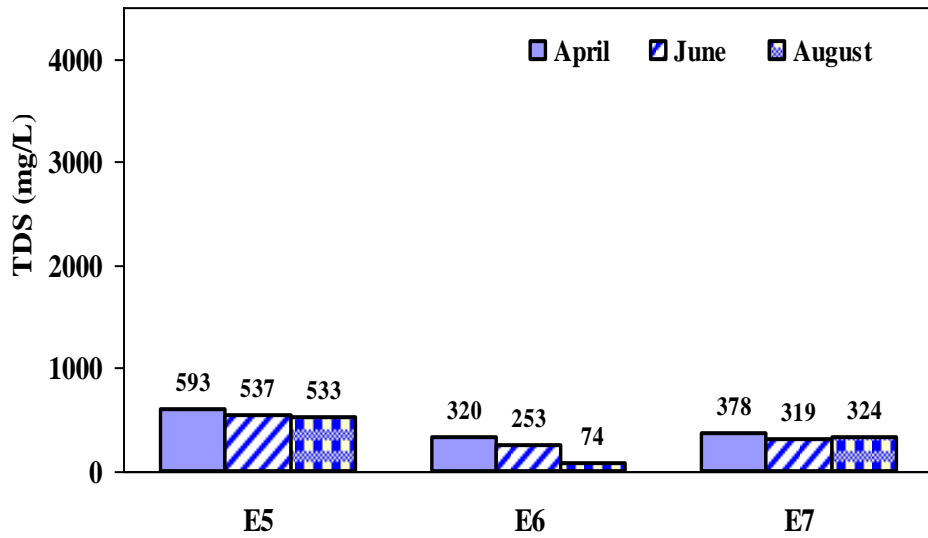


Figure A.87 TDS- Euonymous Potable Water

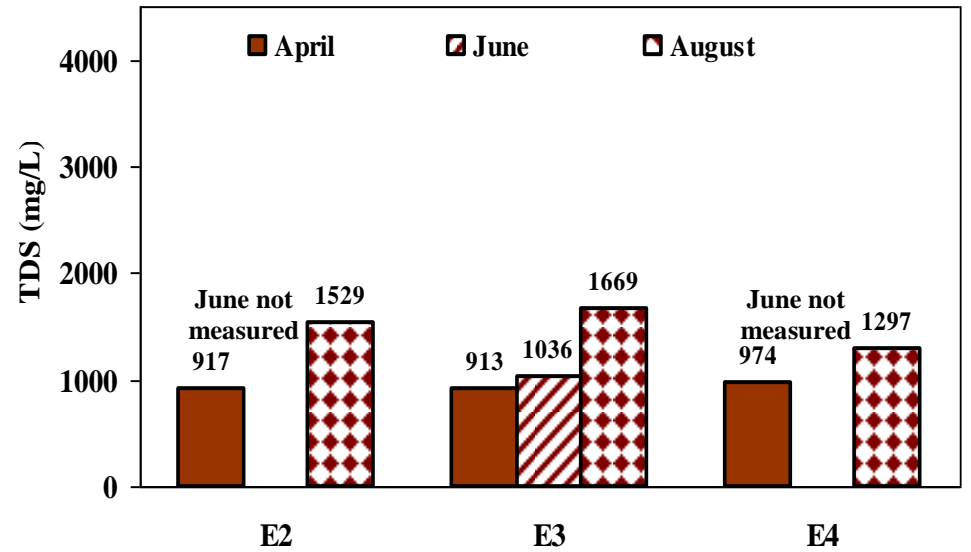


Figure A.88 TDS- Euonymous Graywater

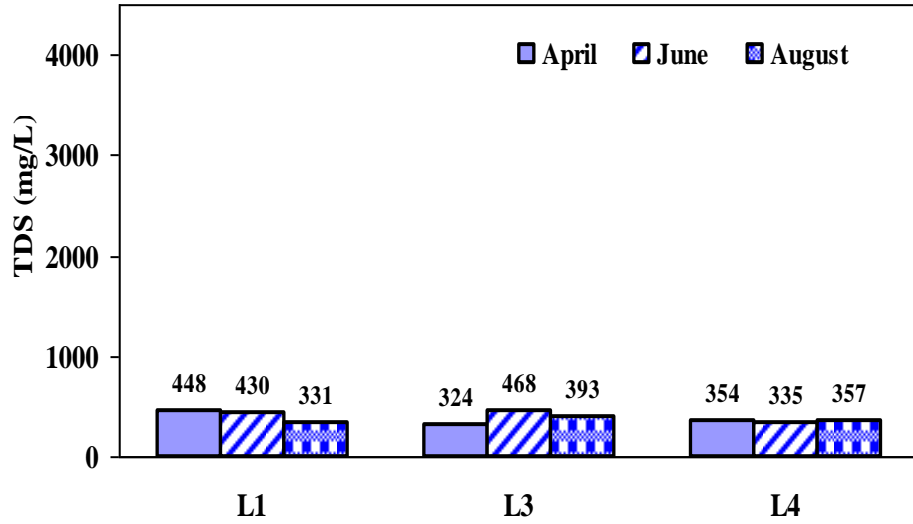


Figure A.89 TDS- Lemon Potable Water

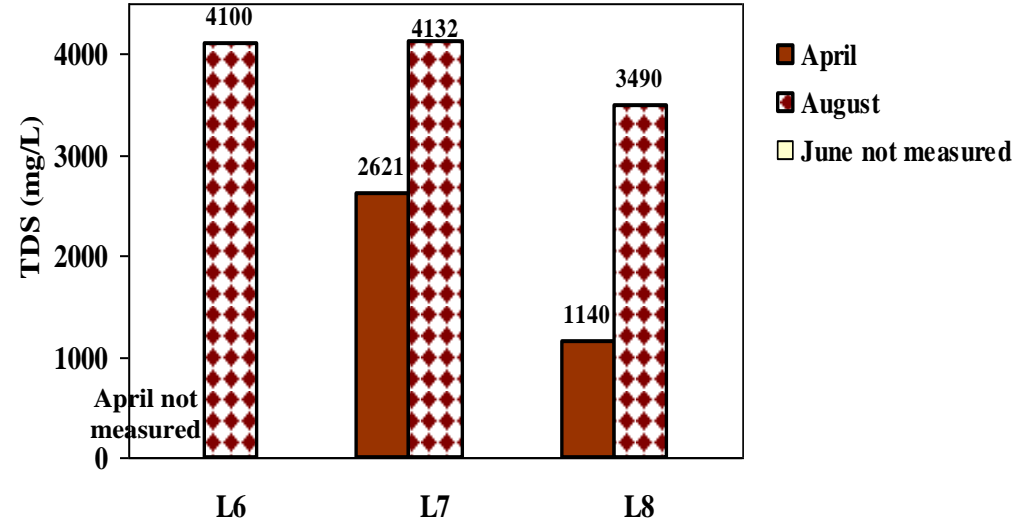


Figure A.90 TDS- Lemon Graywater

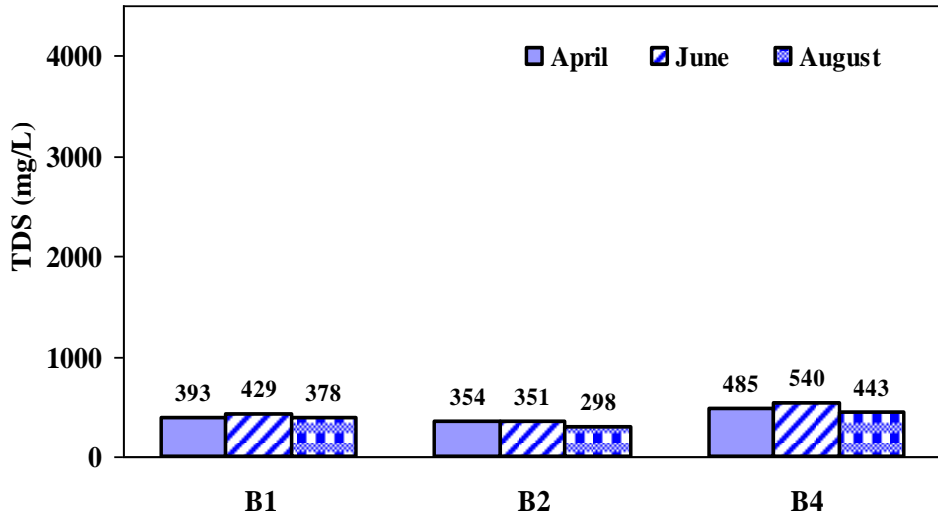


Figure A.91 TDS- Bermudagrass Potable Water

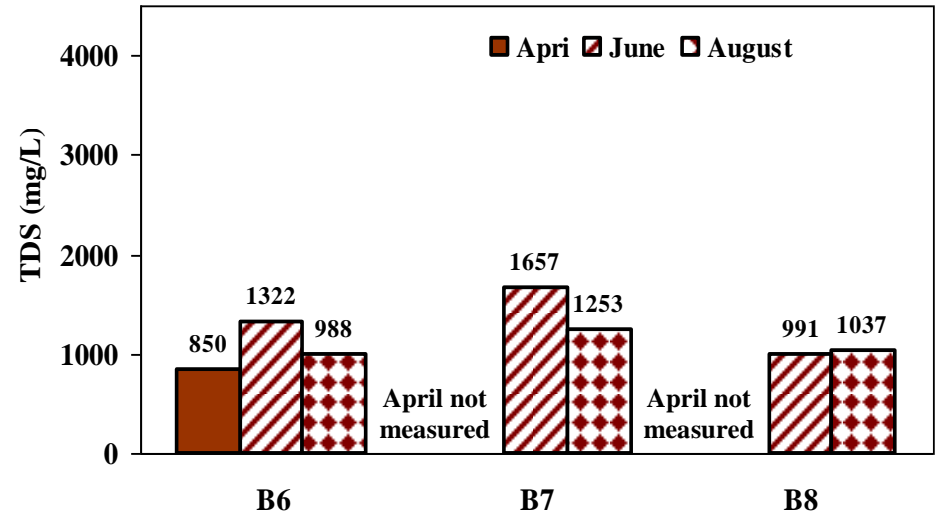


Figure A.92 TDS- Bermudagrass Graywater

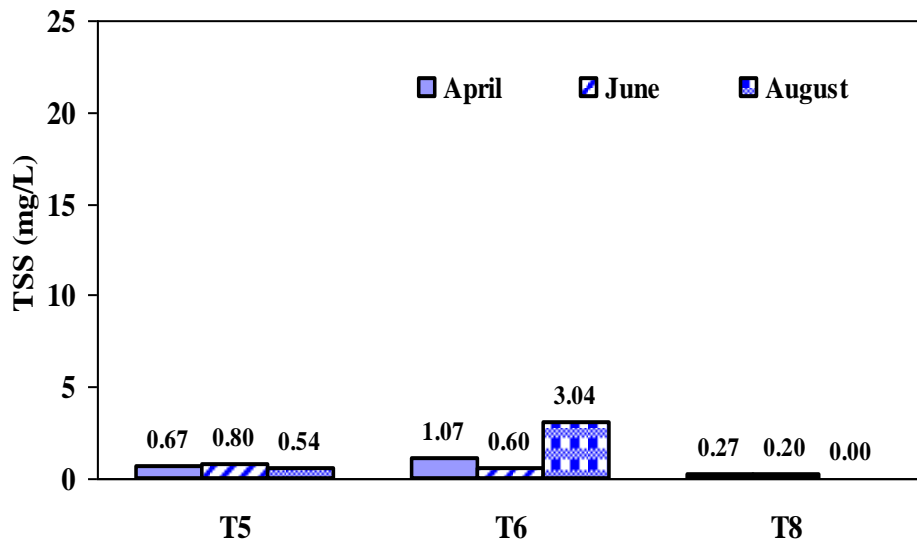


Figure A.93 TSS-Tall Fescue Potable Water

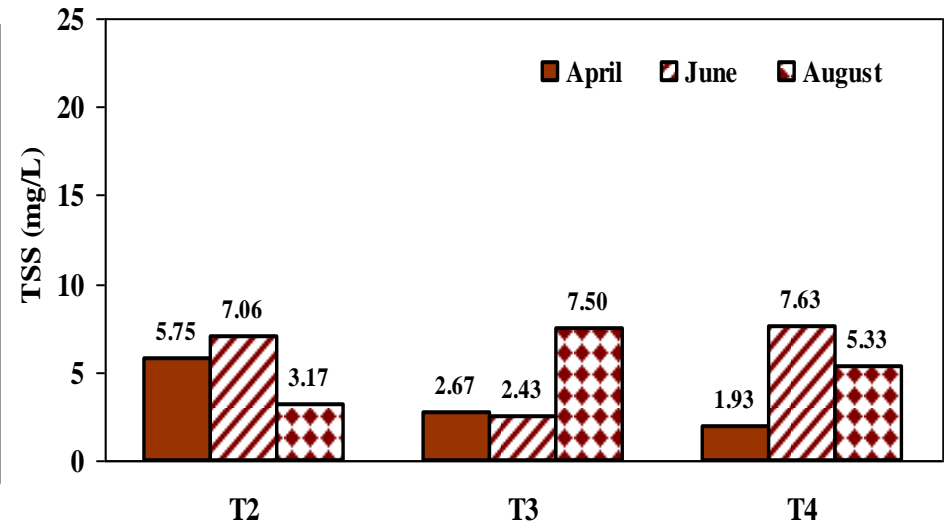


Figure A.94 TSS-Tall Fescue Graywater

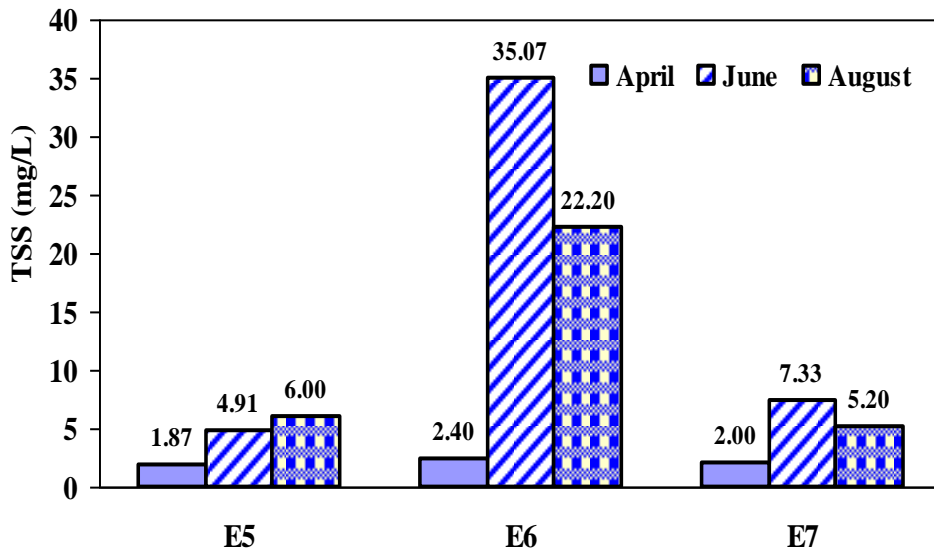


Figure A.95 TSS- Euonymous Potable Water

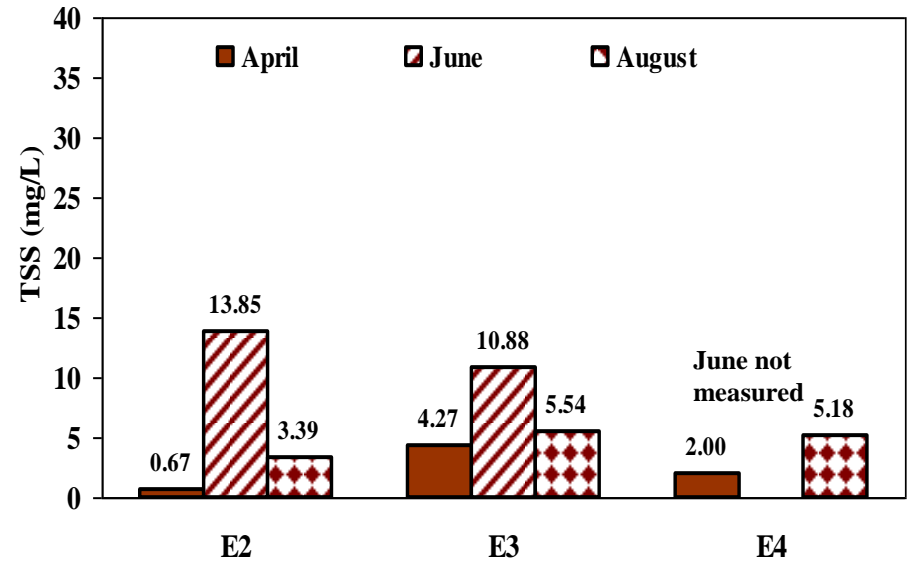


Figure A.96 TSS- Euonymous Graywater

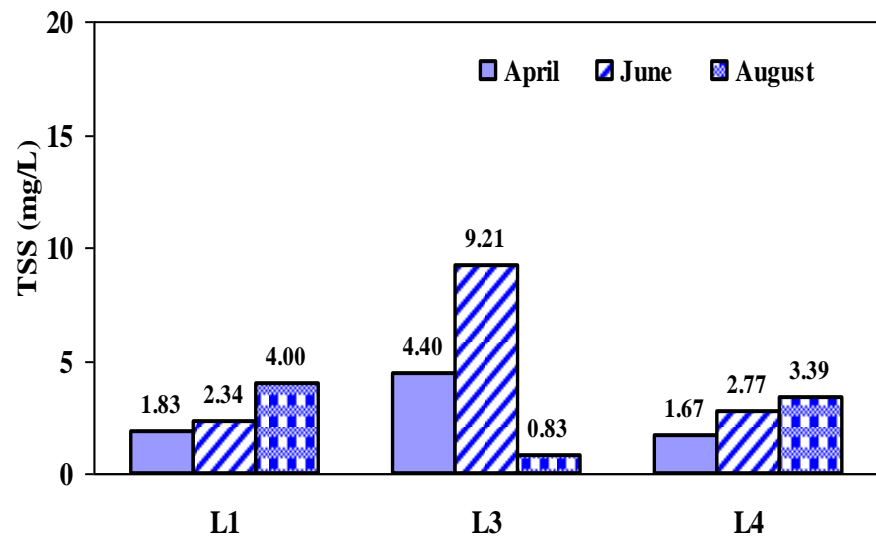


Figure A.97 TSS- Lemon Potable Water

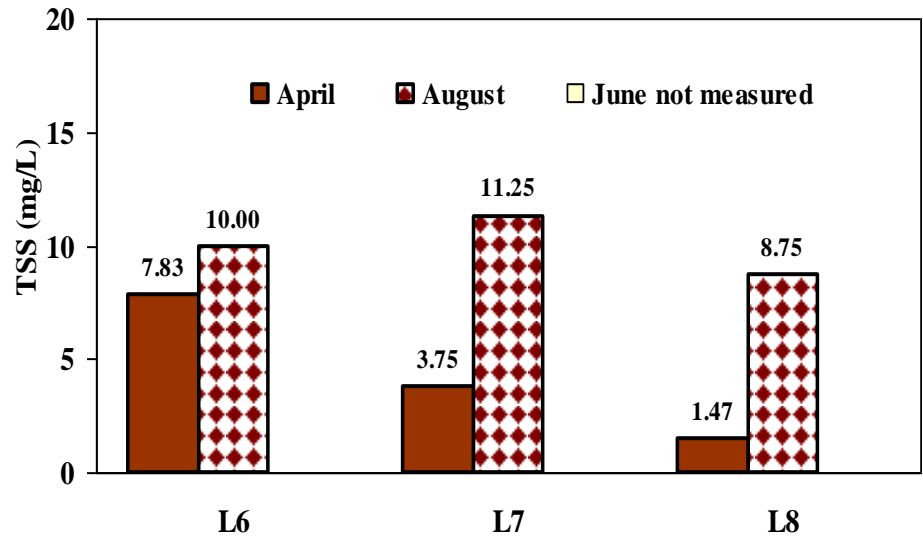


Figure A.98 TSS- Lemon Graywater

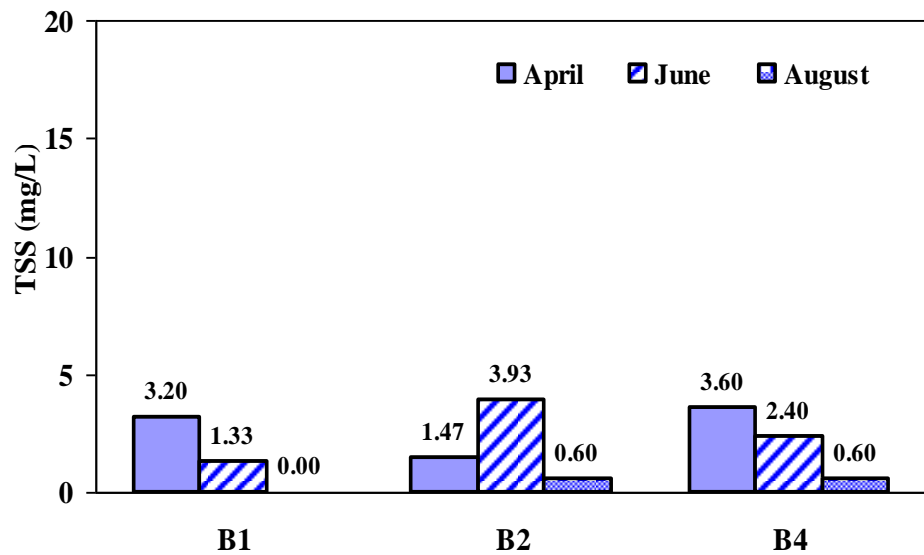


Figure A.99 TSS- Bermudagrass Potable Water

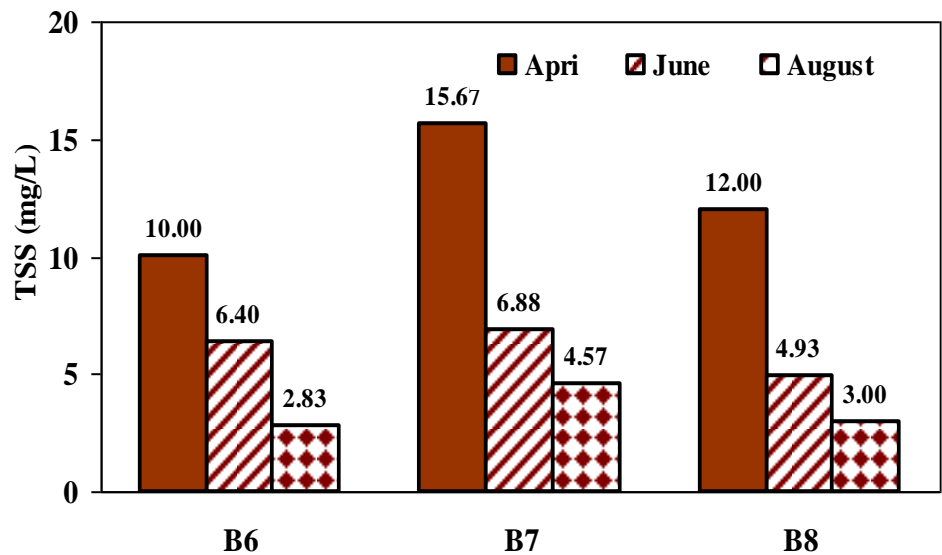


Figure A.100 TSS- Bermudagrass Graywater

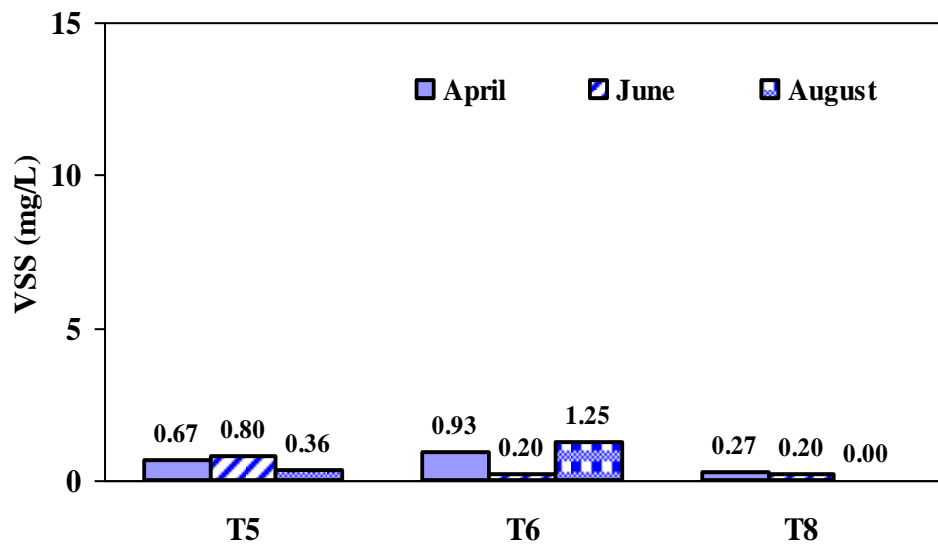


Figure A.101 VSS-Tall Fescue Potable Water

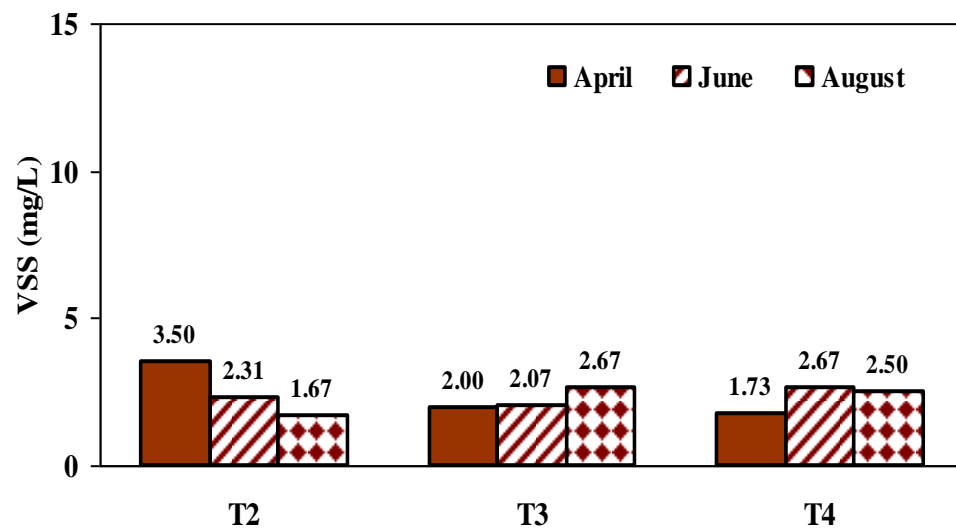


Figure A.102 VSS-Tall Fescue Graywater

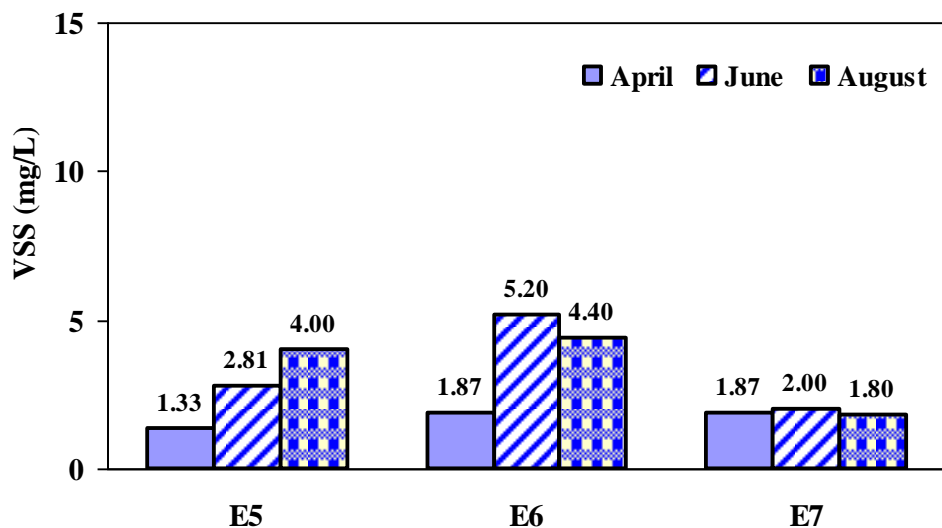


Figure A.103 VSS- Euonymous Potable Water

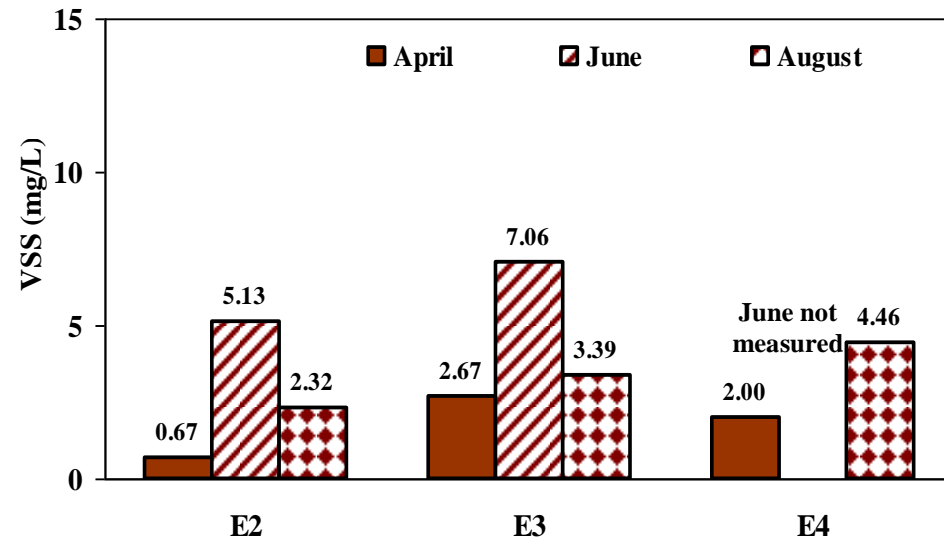


Figure A.104 VSS- Euonymous Graywater

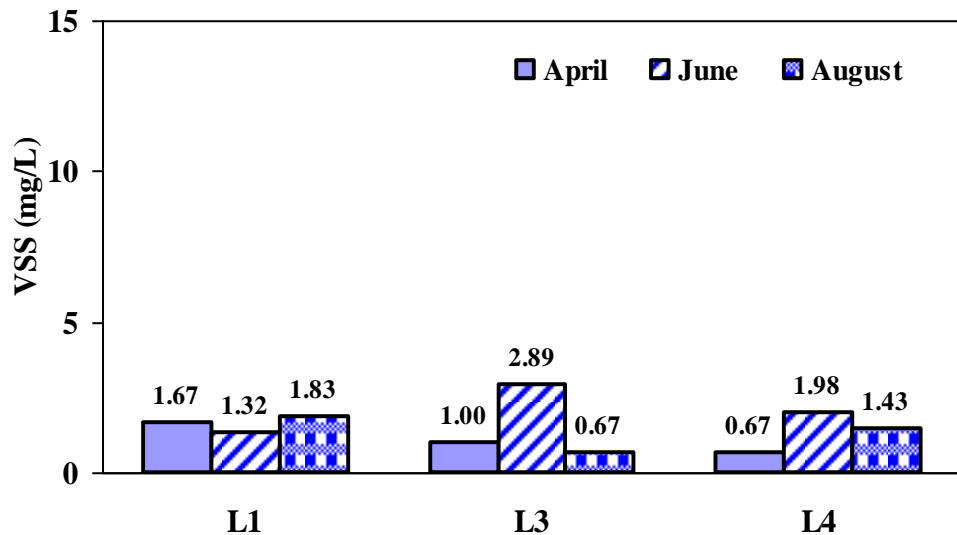


Figure A.105 VSS- Lemon Potable Water

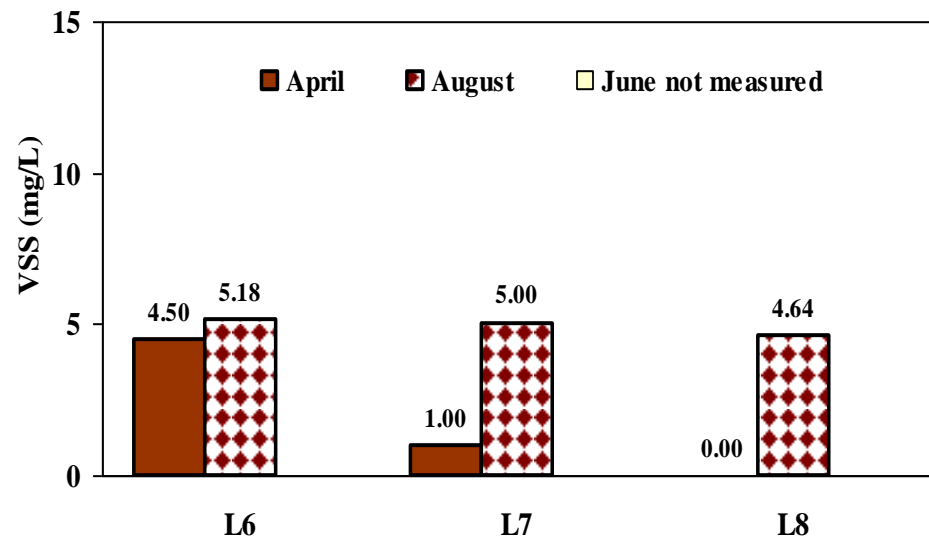


Figure A.106 VSS- Lemon Graywater

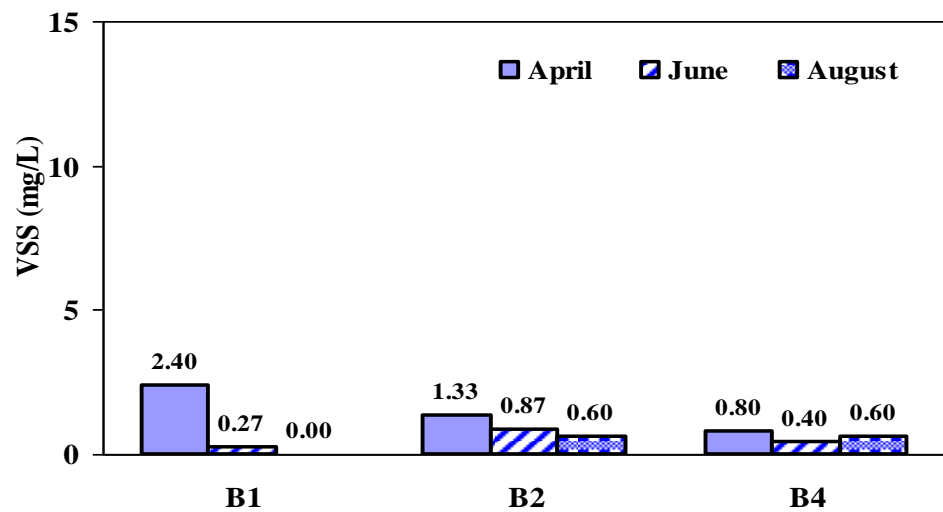


Figure A.107 VSS- Bermudagrass Potable Water

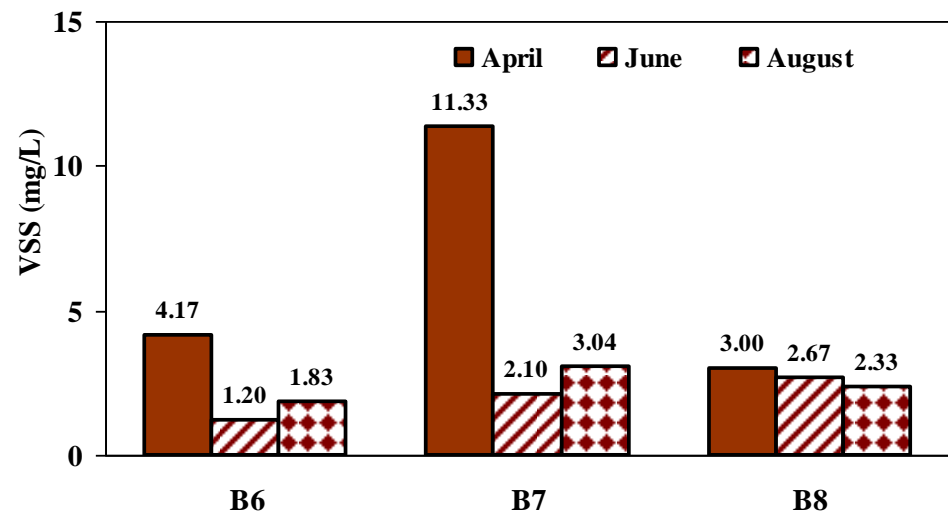


Figure A.108 VSS- Bermudagrass Graywater