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Emergency access to clean water in remote areas, e.g. during disease outbreaks, may be accomplished via portable greywater reuse station, particularly if implemented without need for biological treatment or chemical deliveries. An ozone-UV net-zero greywater system was developed and tested, and is projected to produce water for temporary potable use with 15% rainwater makeup and without generation of infectious concentrate.

Ozone-UV net-zero water wash station for remote emergency response healthcare units: Design, operation, and results

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Abstract

Because disease pandemics can accelerate rapidly in areas with limited clean-water access, a portable greywater reuse system may be useful to provide wash water at emergency health care units. In this study, a novel fed-batch (hybrid continuous-batch flow) net-zero water (NZW), or nearly closed-loop, reuse system comprising screening, 5 μm filter, and ozone-UV advanced oxidation was designed, constructed, and tested for performance with simulated and actual human showers. Water quality was tested for compliance with US drinking water standards, total organic carbon < 0.5 mg/L, and pathogen inactivation including 12 $\log_{10}$ virus, 10 $\log_{10}$ protozoa, and 9 $\log_{10}$ bacteria as has been recommended for direct potable reuse. Energy, operation, and maintenance requirements were also evaluated, along with the system's capacity to handle shock events such as unintentional contamination with urine. Design goals were achieved without the addition of GAC point-of-use filter, except compliance with bromate and nitrate drinking water standards, which were met only for temporary use of up to three years per person. A capacity of 32 showers/day at 1920 W continuous power is projected, without generation of potentially-infectious concentrate. To avoid the further increase in system weight and energy demand needed to address urine input, future integrated urine diversion and collection, and system drain-and-fill following detection of urine in recycled water by electrical conductivity, are suggested for the field unit. Field testing is recommended. Further research should focus on potential need for bromate/nitrate mitigation, and longer-term study of microbiological inactivation.

1. Introduction

The likelihood of a global pandemic is now greater than ever due to population densities and global trade, transportation, migration, and climate change. While early response to infectious outbreaks is therefore essential, recent Ebola crises in Africa highlight the delays in health care response that can result from a lack of running water at remote sites (1). In particular, a critical step in containment of an epidemic is removal of protective gear upon exit from quarantine, and recent CDC guidelines include step-by-step protocol for the task including showering (2). Nevertheless, many health care centers set up in emergency response to the Ebola epidemic lacked running water initially. While Doctors Without Borders/Médecins Sans Frontières generally installs systems, such as wells, that can be used in the longer term, such systems are not feasible in some areas, and a portable system can likely be deployed more quickly, e.g. by the military.

The United States Army (U. S. Army) does employ a portable water treatment unit, the Reverse Osmosis Water Purification Unit (ROWPU). However, the unit requires a continual source of feed water of sufficient quality, and an alternative for continual disposal of both the wastewater generated and a concentrate stream, both containing potentially pathogenic contaminants. Hence, treated water may need to be continually transported to a remote site not having a suitable source water, and concentrate may require treatment prior to disposal (3).

An alternative to single-pass treatment, use, and disposal for emergency response may be a net-zero water (NZW) system having viral inactivation capability. A NZW system has been defined as a water management system that neither imports nor exports significant water to/from the service area (4) or, similarly, a system that "limits the consumption of freshwater resources and returns water back to the same watershed so not to deplete the groundwater and surface water resources of that region in quantity and quality over the course of a year" (U. S. Army, 2015). For example, a portable, nearly closed-loop, direct potable reuse (DPR) wash station could be delivered e.g. by helicopter to an emergency site. In fact, the U. S. Army has a reverse osmosis (RO)-based reuse system for showering, the Shower Water Reuse System (SWRS), comprising microfiltration, RO, and chlorine residual (6,7). However, 25% of the water must be disposed as potentially infectious RO concentrate. Typically, this water is treated by oxidation or disinfection before discharge, requiring additional treatment processes or chemical transport to the site, or treated offsite, requiring transport of the concentrate (8,9). No other portable, small-flow direct potable water reuse systems are known to us (10).

As an alternative to RO, an advanced oxidation-based NZW system can recover 85% of influent wastewater for potable reuse, discharging 15% potable water for irrigation and taking in 15% rainwater makeup, at an energy demand comparable to RO-based systems (4,11,12). Also, at small scales such systems are projected capable of energy-positive operation, saving more energy for heating hot water (due to system retention of hot water thermal energy) than is used in water and wastewater treatment and conveyance combined (4,11,12). The approach involves advanced oxidation of organics to below detection in terms of chemical oxygen demand, without removal of minerals, and has been initially demonstrated to produce water exceeding all drinking water standards and DPR guidelines for chemicals and microbes. However, this approach has been demonstrated only for comingled (black and grey) municipal water management, incorporating biological treatment, coagulation, and ultrafiltration.

The purpose of this study was to design and construct a prototype NZW wash station employing only filtration and advanced oxidation, with RO treatment only of the 15% makeup water stream, and to test its capability to provide wash water under tropical conditions representing deployment and operation at a remote emergency response health care unit. Design objectives included destruction of 10 g/day of organic constituents in 980 L/day (260 gal/day) of water to below 0.5 mg/L TOC, and minimum inactivation of 12-$\log_{10}$ virus, 10 $\log_{10}$ protozoa, and 9 $\log_{10}$ bacteria (13,14) using ≤1300 W of continuous power, or 32 kWh/m^3 (0.12 kWh/gallon) of treated water. The TOC and pathogen inactivation criteria were taken from the

Framework for Direct Potable Reuse and selected for two reasons. First, due to intended system application (infectious disease treatment units), meeting DPR framework standards ensures a high-quality water in an elevated risk application. The second reason for selecting the DPR framework guidelines is that there are currently no guidelines for human contact greywater reuse (10). Additionally, compliance as possible with all potable water quality standards, as required for long-term use (>30 days) by the U. S. Army for shower water (15) in the absence of approval otherwise based on formal risk analysis (7), was desired. The system was designed to operate for a period of at least 1 year with no maintenance. Operational complexity and the need to transport chemicals, fuel, and/or water to the site, with low environmental and spatial footprint (10) and capability to respond to excessive contamination events, such as urination, in real time were evaluated. Results of experiments to characterize ozone-UV kinetics are presented in detail elsewhere (16).

2. Materials and methods

An ozone-UV advanced oxidation net-zero greywater (NZGW) system with shower and sink was designed, constructed, and operated for a period of one year in an open-air parking garage in Miami, FL. Experiments comprising simulated and actual use of the shower, and sampling and analysis of waterborne chemical and microbiological constituents, were conducted to determine compliance with U. S. federal drinking water standards and performance with respect to design goals.

Because sink water was projected to be minor compared with shower water, showers were used in all tests. Several shampoos and soaps were tested for pH and total organic carbon (TOC), as listed in Supplemental Information, Table S1. Goals for soap selection included minimization of organic loading and maintaining near neutral pH in shower water. On those bases, soap was pre-selected as a mixture of Campsuds (Sierra Dawn Products, Graton, CA) mixed with 0.747 M Na_2CO_3 (soda ash). That is, Campsuds was selected based on its low organic loading relative to observed surfactant strength, once diluted 25% for use as a shampoo and soap (Table S1), and the addition of soda ash maintained neutrality in the greywater and treated water, improving kinetics and reducing corrosion (16). For conditioner, Garnier Fructis Biodegradable Conditioner (Garnier LLC, Clark, NJ) was selected due to its neutral pH and claimed biodegradability, when compared with other conditioners (Table S1).

2.1 System Description

A hybrid batch-continuous flow system design, termed here *fed batch* (17), shown in Figure 1, was selected as the final system design, based on initial testing results summarized in the Supplemental Information section S2. The ozone-UV reactor system included a 16-mesh high capacity stainless steel polypropylene-housed t-strainer (McMaster-Carr, Elmhurst, IL) and ozone injection by venturi (dose controlled with electric controller and gas concentration/flow monitor along with manufacturer dissolution graphs), followed immediately by flow through three high-efficiency, low-pressure UV reactors with total 596 W of UV power (NeoTech D338 and NeoTech D438, NeoTech Aqua Solutions, San Diego, CA). Actual UV dose was measured by sensors in the reactor walls. After ozone-UV advanced oxidation, the water passed to a 5 μm fiberglass filter (Graver Stratum, Graver Technologies, Glasgow, DE). Both tanks contained an ozone vent, to allow ozone gas to vent to an outside courtyard. The energy consumption of individual unit processes of the system tested are estimated as follows: 200 W for continuous 28 g/hr ozone generation (corresponding to 25g/hr dissolved ozone dose), 550 W for the oxygen concentrator, 470 W for continuous venturi ozone injection and water recirculation by 0.5-0.75 hp pump (recirculation flow rate of 110 L/min [30 GPM]), and 700 W for the UV reactors and controllers. The energy consumption of other system components, such as the tank mixer and shower pump, were negligible in comparison. The molar O_3:TOC ratio was 0.5 at maximum TOC loading, while the O_3:UV ratio was selected as 0.18, based on experimental kinetic data summarized in a separate study (16). Due to the subtropical Miami climate and UV heating, the shower was run continuously between experiments for evaporative cooling, to maintain a comfortable shower temperature.

In the fed batch design of Figure 1, the greywater tank has a working volume of 227-530 L (60-140 gallons), while the treated water tank has a working volume of 0-303 L (0-80 gallons). Clean water in the treated water tank is used for showers until the volume is used up. Shower water drains to the greywater tank, initially containing 227 L (60 gallons) of previously-treated water, providing dilution of organics to a favorable concentration for ozone-UV treatment. During this time valve V1 (Figure 1) is open and V2 (Figure 1) is closed. As showers progress, treatment of the greywater tank continues until the treated water tank is exhausted. Then, treatment of the greywater tank is continued for an additional time interval to meet treatment objectives, after which V1 will close and V2 will open to refill the treated tank with clean water.

This system was operated at an 85% recycle rate across all experiments, with 18-23% makeup water comprising RO-treated county water (StealthRO200, Hydrologic Purification Systems, CA, USA) and disposal to sewer of 15% treated water after 3-8% evaporative loss. In the field system, the excess may be disposed onsite as a treated irrigation water, to replenish local hydrology from which the makeup water would likely be drawn. An 85% recycle rate was selected for control of excess mineral accumulation, based on prior experience (11,12), because minerals are not substantially removed in such an advanced oxidation-based NZW system. Water was not replaced between experiments. Alternatives to RO makeup water are outlined in subsequent sections. Regarding human subjects, this project followed from a much larger research project in which a net-zero water system was designed, constructed and operated for two years, providing water to a University residence hall apartment for all uses except drinking and cooking. That project was ruled exempt by the University Institutional Review Board because it did not involve collecting data on human subjects, i.e. did not involve collecting data containing any of the 18 specific identifiers noted in the privacy Rule (USA, 45 CFR 46). Likewise, this project involved no collection of data on human subjects; nevertheless, informed consent was obtained from participants taking showers in the system.

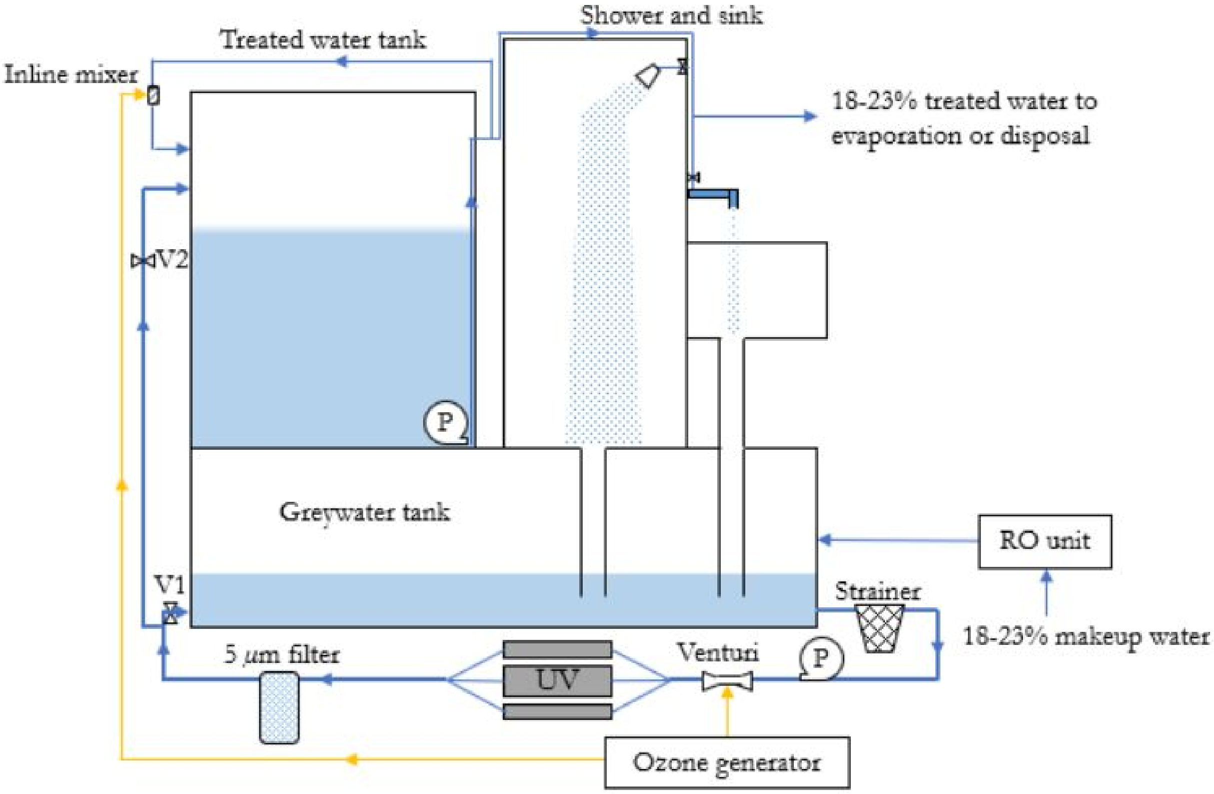


Figure 1. Schematic diagram of fed batch wash station.

2.2 Experiments

TOC was monitored in the treated and greywater tanks, along with other parameters as described subsequently in this section. In fed batch mode, tests comprised a total of 72 showers over a period of one month, with sampling from the greywater tank during and following showers at intervals of 15 minutes during the first and last two hours of sampling, and 30 minutes in between. Initial greywater tank dilution water was 227 L (60 gallons), and eight 10-minute showers were scheduled at consecutive 15-minute intervals. All showers were 38 L (10 gallon) volume, by setting showerhead flowrate to 3.8 L/min (1.0 GPM) and shower duration to 10 minutes. Hence, a total volume of 530 L (140 gallons) was accumulated, and this water was treated for an additional 4 hours after showers concluded. Experiments were performed with and without a GAC point-of-use filter on the showerhead. Samples were collected for potable water analysis, as described in the Supplemental Information (Section S1).

After establishing kinetics of mineralization of greywater, reported in a separate study (16), oxidative kinetics by the fed batch system were tested for showers which included a urination event. In addition, urine detection experiments were conducted, involving 12 showers, 6 with urine and 6 without, during which electrical conductivity was recorded at 15-second intervals over the period of each 9 to 10-minute shower. Urine for these experiments was generated onsite during the showers by the shower participants. The conductivity probe for these experiments was placed in the shower drain trap for continuous measurement.

In initial experiments, the amount of soap required for a shower was found to be significantly different between genders. For men, 10 mL of 25% Campsuds/soda ash solution was found sufficient to achieve a shower than left subjects feeling clean, and was used, along with 1.23 mL of Garnier Fructis conditioner. For women, 25 mL of the 25% Campsuds/soda ash solution was required, along with 4.93 mL of the Garnier Fructis conditioner. In the experiments, an equal number of men and women were selected for showers, to provide an average loading scenario. Finally, the system was spiked with microbes PhiX174, MS2, Phi6, and *B. atrophaeus*, to determine the single pass microbial inactivation kinetics. Sampling methods are described in detail in the Supplemental Information (Section S1).

2.3 Analytical Methods

All chemicals used for analysis were analytical grade and used as received. Total nitrogen, nitrate, nitrite, ammonia, and TOC were measured colorimetrically (UV-Vis spectrophotometer model DU720, Beckman Coulter, Brea, CA) using Hach total nitrogen method 10071 (low range), Hach nitrate method 8039, Hach nitrite method 8507, Hach ammonia method 10031, and Hach TOC method 1029 (low range). Calibration curves were linear and verified bimonthly. Organic nitrogen was found by difference. Hydrogen peroxide was measured by iodometric titration (18). UV_{254} was measured spectrophotometrically (model DU720, Beckman Coulter, Brea, CA) by standard method 5910. pH, electrical conductivity, and dissolved oxygen were measured using an Orion Star A3295 probe unit (Thermo Scientific, MA, USA). Oxidation-reduction potential (ORP) was measured using a sensor in the greywater tank (Analytical Technology, Inc., PA, USA). UV intensity was measured by a sensor in each reactor. All chemical reagents and standards were obtained from Hach (Hach Co., Loveland, CO, USA), with the exception of the hydrogen peroxide titration reagents (Thermo Fisher Scientific, NJ, USA; MilliporeSigma, MO, USA), the soda ash mixed into the soap (VWR International, Radnor, PA), and the *E. coli* analysis agar and membrane filters (MilliporeSigma, MO, USA).

PhiX174 and MS2 are somatic and F+ coliphages, respectively, and were employed to evaluate removal of enteric viral pathogens. These coliphages are routinely used to indicate enteric virus removal during wastewater treatment and log removal values for various treatment processes are summarized in several recent reviews (19–21). Culture stocks of MS2 (ATCC#15597-B1) and PhiX174 (ATCC#13706-B1) were prepared by adding 0.5 ml of coliphage stock to 30 ml of tryptic soy broth (TSB, Fisher Scientific, Waltham, MA) and combining the diluted coliphage suspension with 6ml of the appropriate *E. coli* host (CN-13: ATCC#700609 for PhiX174 or F_{amp}: ATCC#700891 for MS2) in mid-log growth phase and 90 ml of 0.7% tryptic soy agar (TSA) supplemented with appropriate antibiotics (0.015 mg each streptomycin and ampicillin per ml of agar for *E. coli* F_{amp} and 0.01 mg nalidxic acid per ml of agar for *E. coli* CN-13). The suspension was mixed and 4 ml was overlaid onto 1.5% TSA supplemented with antibiotics as stated above in 100 mm plates. After the top agar set, the plates were incubated at 37°C for 24 hours. Coliphages were harvested by adding 5 ml of TSB to each plate and incubating at room temperature for 2 hours. Plates were swirled 20 times and the broth was extracted and combined from all agar plates. The broth was centrifuged at 3000 x g for 5 minutes and the supernatant was filtered through a 0.2 μm filter. Coliphages in stock preparations and experimental samples (spiked water entering the wash station and treated water passed through the ultrafilter) were enumerated using 1 ml of sample (in duplicate) and the double agar layer procedure outline in Standard Methods 9224 (22).

Bacteriophage Phi6 has been evaluated as a surrogate to assess persistence and inactivation of enveloped human pathogenic viruses, like H5N1 avian influenza (23) and Ebola virus (Aquino de Carvalho et al., 2017). The bacteriophage Phi6 and its host *Pseudomonas syringae* LM2489 were obtained from Noreen Adcock (US EPA, Office of Research and Development, Cincinnati, OH). Culture stocks were prepared using the procedures described above for coliphages, with some modification. *P. syringae* LM2489 was grown in tryptone broth with 0.01% (w/v) magnesium chloride and 20 μg ampicillin per milliliter broth to mid-$\log_{10}$ phase, tryptone agar supplemented as above was used instead of TSA and overlaid plates were incubated at 26°C. Harvested Phi6 and experimental samples were enumerated using 1 ml of sample (in duplicate) and the double agar layer procedure (22).

Bacillus endospores have been used in treatment studies since they are tolerant to a wide range of disinfectants. *B. atrophaeus* (ATCC#9372) spore suspensions were obtained from Mesa Laboratories (Omaha, NE). Titers of spores in replicate 1 ml experimental samples were determined using the membrane filtration method outlined in Standard Method 9218B (22).

The concentrations of PhiX174, MS2, Phi6 and *B. atrophaeus* spores in the treated wash station water were determined after elution of the Rexeed 25S ultrafilters (method described in Supplemental Information). Elution consisted of circulation of 400 ml of a filter sterilized solution of 0.01% (w/v) sodium polyphosphate (Sigma-Aldrich, St. Louis, MO), 0.01% (v/v) Tween-80 (Sigma-Aldrich) and 0.001% (v/v) Antifoam Y-30 Emulsion (Sigma-Aldrich) for 2 cycles of 1 min each in a clockwise and counterclockwise direction. The entire volume was recovered and a duplicate 1 ml volumes of the 400 ml eluted sample was used to quantify the spiked microbes. The relative loss of PhiX174, MS2, Phi6 and *B. atrophaeus* spores by filtration and elution with the ultrafilter was evaluated in the laboratory by spiking the microbes in dechlorinated tap water and processing the samples (n=2) as described. The concentration of each microbe in concentrated treated wash water was determined by multiplying the average of duplicate measurements of CFU/PFU per ml by a factor of 10,000 (10000 ml of total wash water) and 1/proportion of microbe recovered (recovery efficiency) to determine the total microbial load in the volume of water passed through the treatment system. The reduction of microbes across the treatment train of the wash station was calculated using the formula $\text{Log}_{10}\ (N_X/N_0)$, where N_X is the microbe concentration after treatment and N_0 is the microbe concentration before treatment. In cases where concentrated treated effluent water samples result in no measured microbes, a value of 2.6 $\log_{10}$ PFU or CFU will be used for N_X as this accounts for the limit of quantification of the culture assays (the detection limit for each culture assay is 1 PFU or CFU/ml and only 2 ml of the 400 ml sample elution volume was analyzed, potentially leaving 398 CFU or PFU in the remaining 398 ml of concentrated sample).

2.4 Modeling and Statistics

To develop the initial system design, preliminary pseudo-first order modeling was conducted assuming nearly complete UV photolysis of dissolved ozone [molar extinction coefficient 2 orders higher than that of TOC (25)] and an initially assumed molar ratio ozone dose:maximum TOC loading of ~0.25 based on design objectives. Subsequently, a previously developed second order model for the ozone-UV process was applied to determine the additional treatment time required for urine organics (16). The only modification to the previously developed model involved separating the urine organics from the typical greywater organics (~18.4% urine organics, based on the difference in maximum TOC between runs with and without urine) and using a separate second order rate constant for each. Steady state mass balance modeling of nitrogen, bromide, and TDS was also conducted based on measured and calculated concentrations of each in soap, conditioner, and body washings. Average measured concentrations of each compound were assumed as input for each shower, with 15% of the daily flow rate of shower water containing the bromate, nitrate, and TDS discharged to the environment. Corresponding replacement of 15% of the flow with RO-treated water was assumed to contain negligible bromide, nitrogen, and TDS.

For disinfection modeling, Chick's Law was used (26). To obtain system specific conservative disinfection values, ozone contact time was assumed equal only to the travel time between ozone injector and the UV reactor, estimated for the 2-inch pipe at 4.0 seconds, and available UV radiation was assumed equal to 8 mW/cm^2, the minimum observed during tests with maximum organic (and ozone and hydrogen peroxide) loading. Calculations were based on the number of times the treatment tank cycled through the recirculation system, beginning after showers had concluded and continuing until the end of treatment.

All error bars and bounds in plots and text represent one standard deviation, unless otherwise noted, to account for the relatively large variation in measured concentrations across experimental runs, attributed to variation in the level of cleanliness of subjects prior to showering. For statistical data comparison, *t*-tests were used (tested for $p < 0.05$). Using Microsoft Excel 2016, data was first verified normally distributed using the chi-squared goodness of fit test, then Levene's test was used to assess variance equality (27). Afterwards, the *t*-test was performed.

3. Results

3.1 Organic Loading and Greywater Parameters

Analysis of the greywater generated by the study population (25 people, 12 male and 13 female) for TOC and routine water quality parameters was performed, with results shown in Table 1. *T*-tests rejected any significant difference between the turbidity, dissolved oxygen, and temperature levels in greywater generated by males and females ($p > 0.05$). However, *t*-tests performed on data observed for pH, UV_{254}, and electrical conductivity indicated significant differences ($p < 0.001$). This result was attributed to the amount of soap mixture used, which was 2.5 times higher for females than for males.

Based on the data of Supplemental Information Table S1 and the shower conditions tested, TOC added as soap was 375 mg/shower for men and 937.5 mg/shower for women, while TOC added as conditioner was 269 mg for men and 1080 mg for women. TOC added as body organics was found by mass balance. Males added 933 ± 451 mg of TOC per shower and females added 1323 ± 675 mg of TOC per shower. In both cases, body organics input was a significant portion of TOC loading. A *T*-test rejected significant difference between male and female body organics that washed off ($p = 0.106$). Also of note, shower water temperature during system testing was too hot for showering (~45°C) due to the subtropical climate and heat input from the treatment system. Hence, the showerhead was kept running between experiments for evaporative cooling, resulting in the loss of 400-700 W of heat energy in the water.

Table 1. Average of Greywater Parameters Over 25 Samples (12 male, 13 female)

Parameter	Overall Average	Standard Dev.	Male Average	Standard Dev.	Female Average	Standard Dev.
TOC (mg/L)	56.5	22.4	39.5	11.9	72.1	17.9
Turbidity (NTU)	19.8	14.3	14.0	8.6	25.0	16.7
pH	8.36	0.69	7.76	0.49	8.92	0.16
Dissolved O_2 (mg/L)	9.88	0.49	9.93	0.58	9.83	0.40
Temp (°C)	36.6	1.39	36.5	1.56	36.7	1.27
UV_{254} (cm^{-1})	0.229	0.092	0.181	0.067	0.273	0.092
Electrical conductivity (μS/cm)	91.9	27.8	65.6	16.3	109	17.0

3.2 Fed batch system organics mineralization

The kinetics of organics mineralization by the fed-batch design, with and without a final GAC polishing step, are reported in detail elsewhere (16). A summary of the results of three 6-hour runs of 8 showers (scheduled as described in the Methods section) with an even number of males and females, are shown in Table 2. As shown, the insertion of the point-of-use GAC filter at the showerhead to polish the treated water had little effect on total organics. In either case, organic load was reduced below the target 0.5 mg/L TOC by the end of treatment, i.e. within a total of six hours including the two-hour period of showering (and prior to refill of the treated water tank).

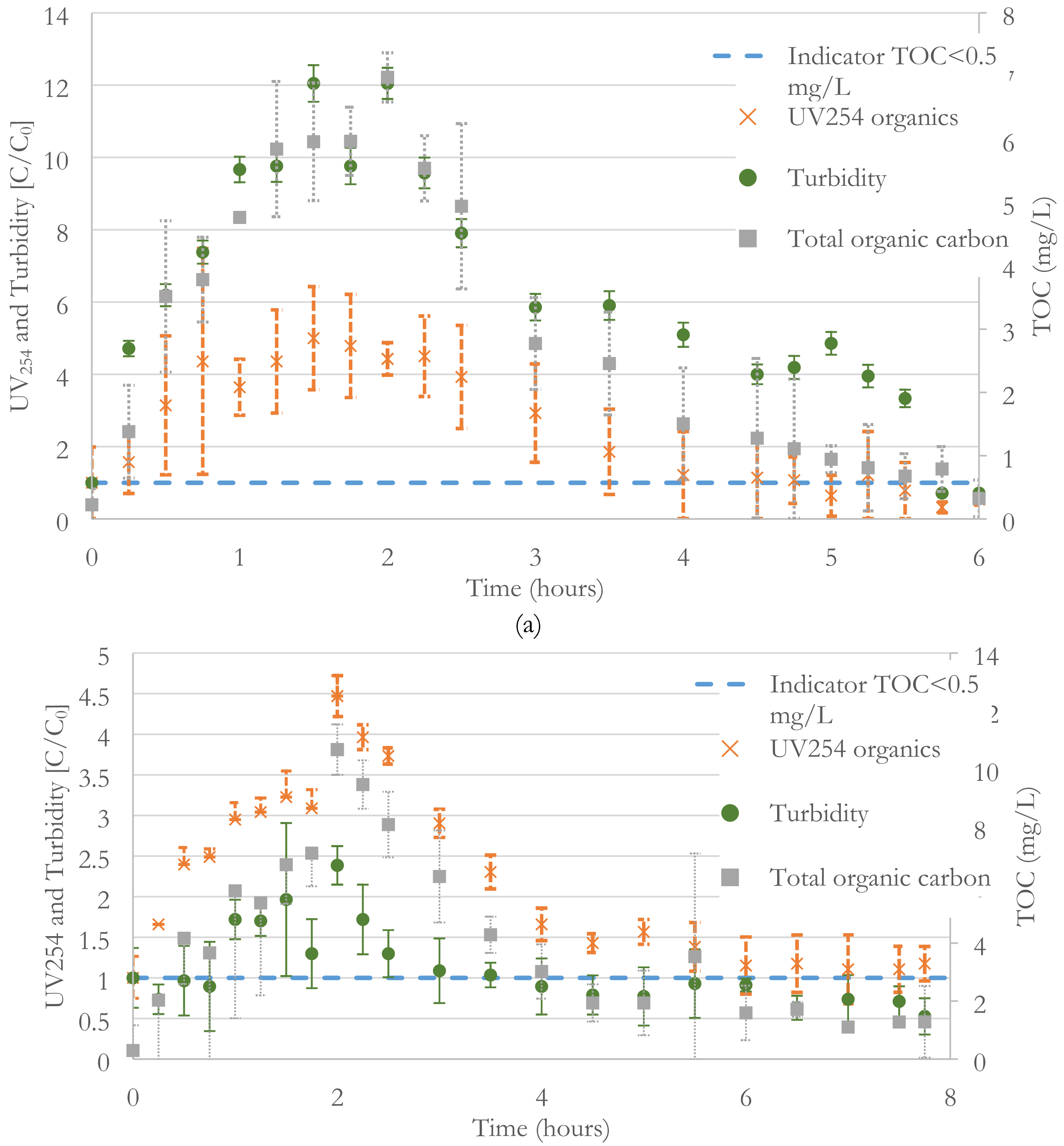
Based on results of kinetic modeling presented elsewhere (16), a system capacity of 1.3 showers/hour was found for the tested sequence of showers followed by continued treatment. This capacity would fall to 0.68 showers/hour for the case of equally-spaced showers, as each shower would require 88 minutes of treatment before the next shower (16). Second order rate constant for TOC mineralization by hydroxyl radical was observed at $[2.0 \pm 0.35] * 10^7$ $M^{-1}s^{-1}$ for the four-hour treatment period following showers (16). Electrical energy per order of magnitude TOC removal per cubic meter of water (EEO) values are also assessed under varying mineralization scenarios in the separate study. For this particular system, EEO (electrical energy consumed per order of magnitude TOC removal per cubic meter of treated water) was projected to be 4.42 kWh/order/m^3 in field conditions (16).

Table 2. Experimental Results for the Fed Batch System (Samples Taken in the Greywater Treatment Tank)

Run	Maximum initial TOC (mg/L)	Standard deviation initial TOC (mg/L)	Mean TOC after 6 hours treatment (mg/L)
GAC	8.71	1.34	<0.35
Non-GAC	6.98	0.39	<0.35

3.3 End of Treatment Indicators

For the experiments described in this paper (10-minute showers every 15 minutes for two hours), a four-hour treatment time following showers was sufficient to achieve disinfection goals and TOC < 0.5 mg/L. However, this after shower treatment time will change with shower frequency in a fed batch system design. Hence, turbidity and UV_{254}, the latter of which may be read approximately on the UV intensity meter associated with many UV reactors, were tested as inexpensive in-line monitoring alternatives to TOC. These parameters, measured over the treatment period for showers (a) with and (b) without a urination event, are shown in Figure 2 as a C/C_0 ratio on the left axes, while actual TOC concentrations are plotted on the right axes. Hence, a value <1 indicates TOC <0.5 mg/L if the indicator accurately represents TOC. In Figure 5(a), UV_{254} appears adequate for determining the end of treatment in the absence of user urination in the shower, while turbidity appeared to be unreliable between the two run types. ORP, previously reported to signal the end of peroxone treatment (28), was ineffective for the process reported here, actually increasing with soap addition and stabilizing during treatment. Also, although TOC <0.5 mg/L was not achieved in the presence of urine [Figure 2(b)], none of the indicators tested appeared to have a reliable relationship with TOC in that case, perhaps due to significant differences in the urine organic matrix. Urination is discussed further in subsequent sections.



(b)

Figure 2. Potential end of treatment indicators for the fed batch runs (a) without urine and (b) with one urination event. [Initial conditions (a): 178 µS/cm electrical conductivity, pH 6.87, UV_{254}(TOC < 0.5 mg/L) = 0.01cm^{-1}, turbidity(TOC < 0.5 mg/L) = 0.14 NTU, 16 mg/L DO, 37^{o}C; (b): 189 µS/cm electrical conductivity, pH 7.08, UV_{254}(TOC < 0.5 mg/L) = 0.007 cm^{-1}, turbidity(TOC < 0.5 mg/L) = 0.19 NTU, 16 mg/L DO, 38^{o}C]

3.4 Water Quality Standards

The concentrations of all constituents detected in the analysis for compliance of the treated water with all Florida drinking water standards are listed in Table 3. In the run using the GAC filter, all primary and secondary standards were met, with the exception of nitrate, which was 10.7 mg/L as N (standard is 10 mg/L). Bromate was not detected in this run. However, in the run without the GAC filter, bromate exceeded the standard at 32 μg/L (standard is 10 μg/L), while nitrate exceeded at 14.2 mg/L. Of note, the treated water easily met both drinking water standards calculated proportionally for short-term use of 6 months (based on US EPA exposure factor average 70-year lifespan), i.e., 1400 mg nitrate/L and 1400 bromate μg/L (29). Brominated disinfection byproducts (DBPs) were not detected in the drinking water standards analysis, expected because although they are formed during chlorination of bromide-containing waters (25,30), continuous application of ozone converts all reactive bromide to bromate (31). Hence, bromate was the only byproduct detected in product water, as was also found previously for the municipal net-zero water reuse process which included chlorination (11,12).

Table 3. Concentrations of All Drinking Water Parameters Detected in Treated Net-Zero Water of June 2016, Analyzed by Certified External Laboratory

Parameter	8-Jun-16 GAC	15-Jun-16 Non-GAC	Drinking water standard
	Inorganic (mg/L)		
Antimony	0.00160	0.00152	0.006
Barium	0.00422	0.00483	2.0
Cadmium	ND[1]	0.0000984	0.005
Chromium	0.00227	0.00227	0.1
Lead	ND	0.00013	0.015
Nickel	0.00114	0.00120	-
Nitrate	10.6	14.7	10
Sodium	36.3	44.4	-
	Secondary (mg/L)		
Aluminum	0.0535	0.0413	0.2
Chloride	14.7	17.8	250
Copper	0.0237	0.0139	1.0
Fluoride	0.132	0.0020	2.0
Sulfate	10.3	13.4	250
Zinc	0.911	0.887	5
Color[2]	0/7.34	0/9.55	15
Threshold Odor Number[3]	1.00	1.00	3
Total Dissolved Solids	142	174	500
	DBPs (μg/L)		
Bromate	ND	32	10
Total Haloacetic Acids	ND	ND	60
Total Trihalomethanes	1.05	ND	80
	Other		
Total Coliform	Absent	Absent	5% positive/month

[1] Not detected

[2] Units: Pt-Co

[3] Units: T. O. N.

3.5 Nitrogen species, bromate, and TDS

Based on the results shown in Table 3, tests were performed to identify sources of bromide and nitrogen to the system. The results for nitrogen analysis are presented in Figure 3. The primary source of nitrogen was washings from the human body, mostly in the form of organic nitrogen. Nitrogen addition from the Garnier Fructis conditioner was almost negligible, while nitrogen addition from the soap was lower than from the body. Nitrogen inputs to the RO pretreatment were also assessed, with results for nitrogen in the influent and effluent of the RO system shown in Supplemental Information (Figure S4). The RO system passed a significant amount of total nitrogen in the source water through to the system. Nevertheless, RO units are typically effective for >99.9% nitrogen species removal, though nitrate removal may be significantly lower (32–34). Therefore, some of the nitrogen entering the system may be addressed through adequate pretreatment of the source and makeup water, though nitrogen inputs from the soap and body are also significant.

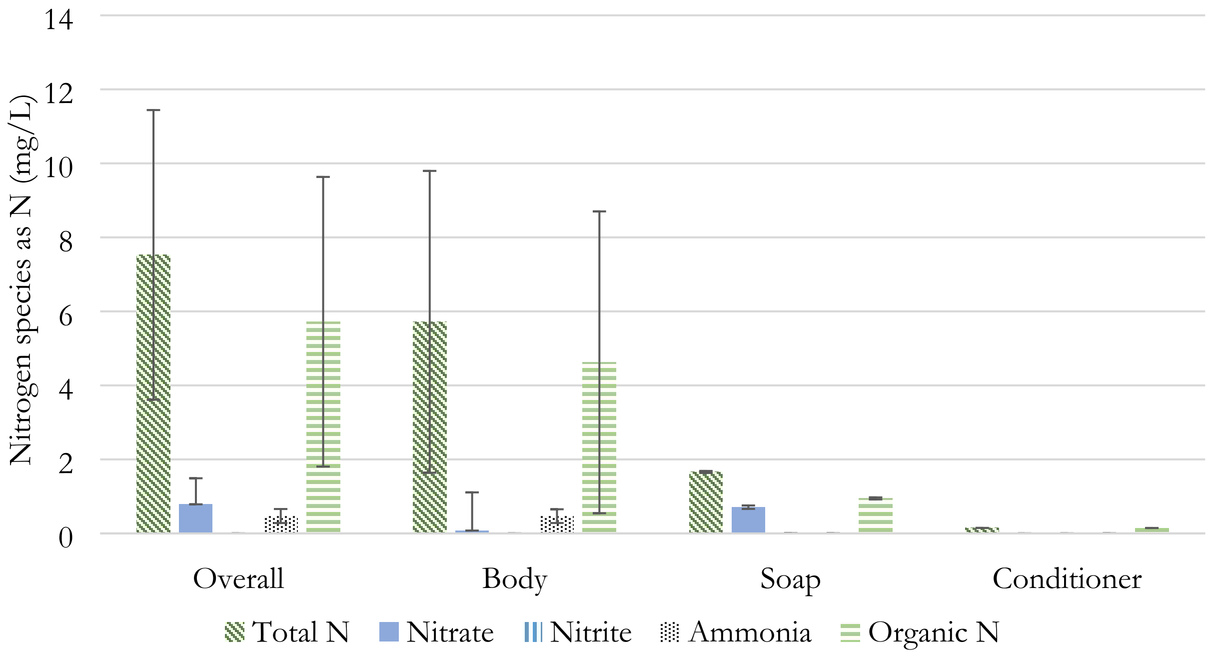


Figure 3. Nitrogen species addition on a per shower basis. [Conditions: 37.8 L shower volume, 25 mL of 25% Campsuds with 0.747 M soda ash, mL of Garnier Fructis biodegradable conditioner, 37°C, 164 µS/cm electrical conductivity]

The results of analysis of bromide entering the system are shown in Supplemental Information (Figure S5). Bromide tested significantly higher in three samples of the RO effluent than in the samples of the influent prior to passing through the RO, on the same day. This result could indicate sample contamination, short-term variability in city water bromide concentration during that time, or contamination of the RO system, as RO units should be effective at retaining bromide (35,36). Total bromide in the greywater samples was measured at the level expected due to input from the shampoo and conditioner used, at 21.7 µg/L. Therefore, given a well-functioning RO system, most of the bromide input would come from the soap and conditioner, with negligible bromide input from the human body.

Steady state concentrations of bromate, nitrate, and TDS that would accumulate in the system were modeled. Inputs are assumed as 7.5 mg/L total nitrogen, 21.7 µg/L bromide, and

55.9 mg/L TDS, per shower, based on greywater sample analysis (Table 1, Figures 3 and S4). Results are shown in Figure 4. Measured concentrations of TDS, bromate, and nitrate, from the water quality standards analysis, taken with point-of-use GAC filters on the showerhead, are shown for comparison. While the TDS points match well with the model results, nitrate is slightly lower than modeled, and bromate does not agree well. Both of these latter results are presumably due to full bromate reduction/adsorption and partial nitrate adsorption by GAC in the preceding tests, and until the GAC was removed, immediately after the first sample. Model results indicate maximum steady state concentrations of bromate, nitrate, and TDS of 232 μg/L, 50.2 mg/L, and 385 mg/L, respectively, assuming an 85% recycle rate. Hence, at an 85% recycle rate, with no complete water changes performed and without GAC polishing, the water would be expected to meet all short-term drinking water standards, adjusted proportionally for a total period of use up to three-years assuming 50% male and 50% female users, (calculated from the 70-y average lifespan, USEPA exposure factor), after which period the bromate concentration would exceed the adjusted standard (233 μg/L).

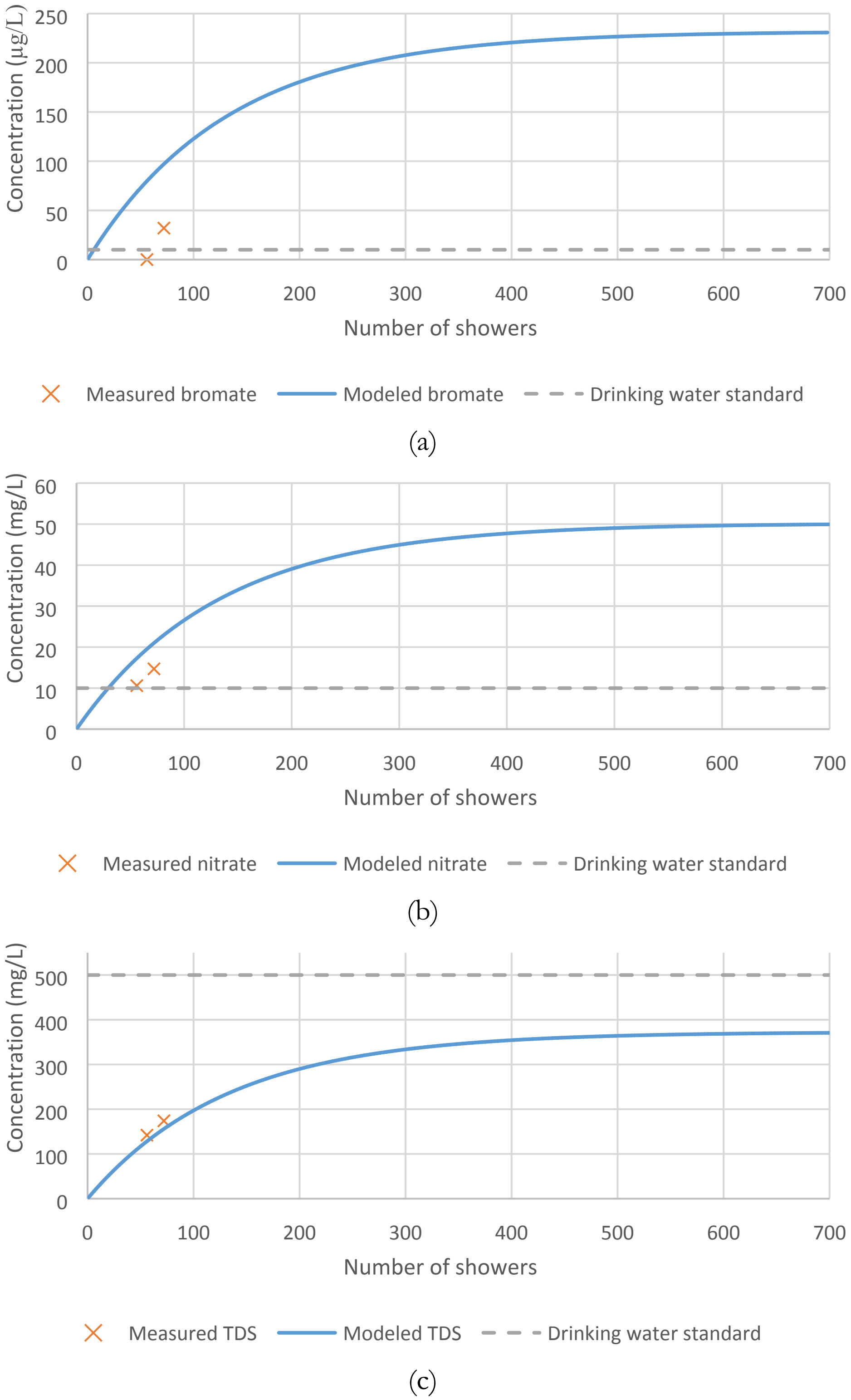


Figure 4. Steady state modeling and results of bromate (a), nitrate (b), and TDS (c) in the system, assuming 85% recycle rate and 99.9% rejection of species in makeup water by RO.

3.6 Microbiological Results

E. coli results, measured at the end of each treatment period in the final sample, were consistently less than 1 CFU/100 mL. Results of single-pass microbiological inactivation tests are shown in Table 4. Total influent concentrations varied from 3.08•10^8 to 6. •10^9 PFU PhiX174, MS2, Phi6 or CFU *B. atrophaeus*. In a single pass, all species were inactivated beyond detection limits, at a UV dose of 596 W and delivered ozone dose of 25 g/hr, with an average intensity reading of 12 mW/cm^2. Therefore, a single treatment pass was found sufficient to inactivate the highest concentration of each species that could reasonably be tested. Performance evaluation of the ultrafiltration and elution method used to measure microbes in concentrated effluent water showed that mean recovery efficiency was 76 (standard deviation = 47), 77 (10), 25 (11) and 124 (96)% for PhiX174, MS2, Phi6 and *B. atrophaeus* spores, respectively. Hence, loss of PhiX174, MS2, Phi6 and *B. atrophaeus* spores during sample processing of treated water is minimal and overall reductions of 6-7 logs can be attributed to the treatment employed.

Table 4. Single pass removal of microbes.

Microbe	Mean Concentration[a] (Log$_{10}$ PFU or CFU)	
	Influent	Effluent
PhiX174	8.6	LOD[b]
MS2	9.7	LOD
Phi6	10.4	LOD
B. atrophaeus	8.8	LOD

[a] Single pass inactivation experiments were performed three times

[b] LOD is limit of detection; LOD is 2.6 log$_{10}$ PFU PhiX174, 2.6 log$_{10}$ PFU MS2, 2.6 log$_{10}$ PFU PhiX6 and 2.6 log$_{10}$ CFU *B. atrophaeus.*

Calculated log$_{10}$ inactivation of MS2, commonly used as a potentially conservative surrogate for viral inactivation due to its non-enveloped structure (13), by the fed batch system was estimated at >17.6 log$_{10}$, based on inactivation rates reviewed previously (10). Calculated log$_{10}$ inactivation for bacterial and protozoa surrogates (total coliforms and *C. parvum*) are 10.6 and 14.0 log$_{10}$, respectively. Results are shown in Table 5. All calculated $\log_{10}$ removals for the full treatment exceed treatment goals for the DPR framework, which are 12 $\log_{10}$ viruses, 10 $\log_{10}$ protozoa, and 9 $\log_{10}$ bacteria (13,14).

Table 5. Estimated system-specific $\log_{10}$ inactivation of microorganisms in treatment processes (10).

Process	Dose	Ct	Calculated $\log_{10}$ inactivation		
			MS2	Total Coliforms	*C. parvum*
UV	8.0 mW/cm^2	76.8 mJ/cm^2	4.7	3.84	5.99
Ozone	3.82 mg/L	15.3 mg-sec/L	10.4	0.01	0.26
5 μm fiberglass filter	-	-	>0.8	>5	>6
Total single pass		-	>15.9	>8.85	>12.25
Total for full treatment		-	>17.6	>10.6	>14.0

3.7 Urine Detection and Kinetics Results

Three runs of 8 showers in the fed batch system were conducted with urination, as previously described, for kinetic analysis. In these experiments, the worst-case scenario was assumed, that the urination event occurred in the last shower before treatment, so that the time available for treatment was minimized. These results are shown in Figure 5. As shown, urination in the shower overloads the system not only in terms of added nitrate and bromate (25,37), but also in terms of organics. In fact, whereas urea biodegrades readily (38,39), its second order rate constant for reaction with •OH has been reported at only 7.90×10^5 $M^{-1}s^{-1}$ (25). Hence, the rate of oxidation was much slower with urine addition.

The second order rate constant for reaction of urine organics with hydroxyl radical was determined to be 1.3×10^6 $M^{-1}s^{-1}$ by the modeling process, nearly double the literature value of urea's second order rate constant, presumably due to organics other than urea in the urine being more easily oxidized. This rate constant is an order of magnitude lower than the rate constant for reaction of greywater organics with hydroxyl radical, 2.0*10^7 $M^{-1}s^{-1}$ (16). Thus, the modeled time required to reach a TOC concentration of 0.5 mg/L was 9.7 hours for the case shown, compared to 6 hours without urination, and could be significantly more with additional urination events. When considered along with nitrate and bromate concentrations, urination might be addressed via integrated male/female urinal equipment. Further, system drain-and-fill would be required in case of urination to the shower drain or other excessive stress on the treatment system, such as organic or solids overload from misuse.

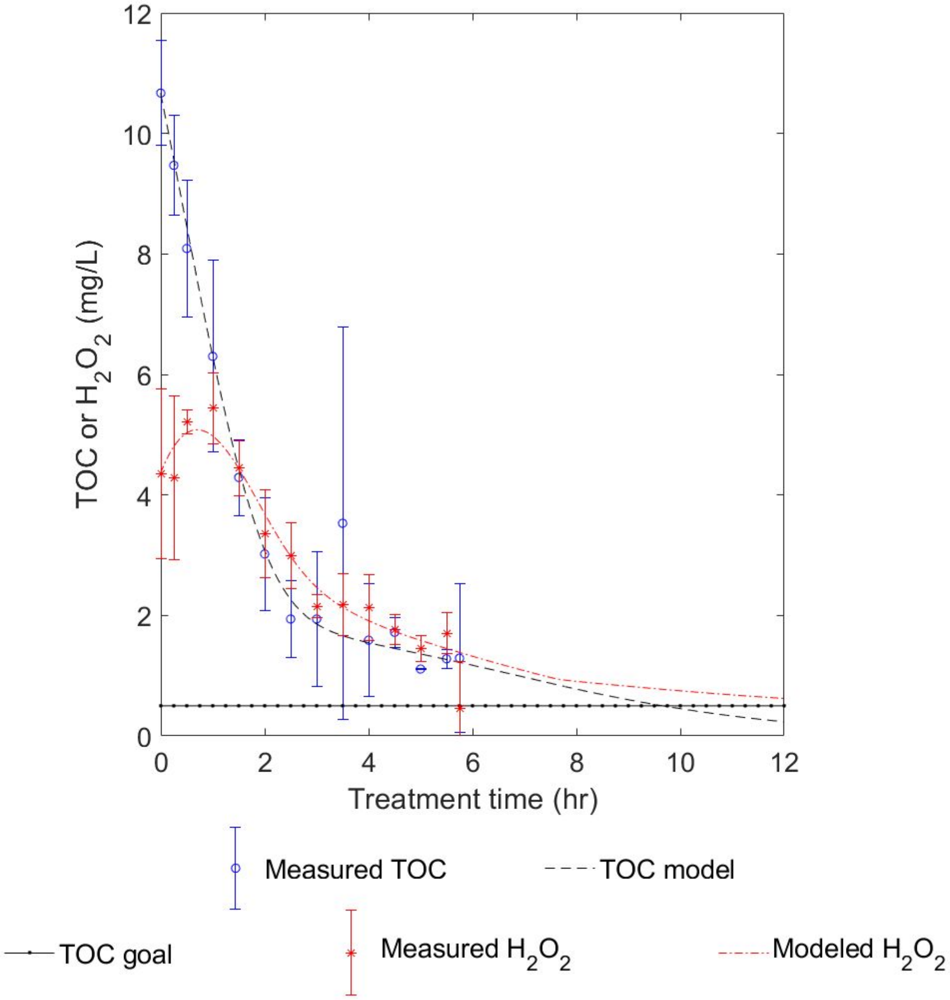
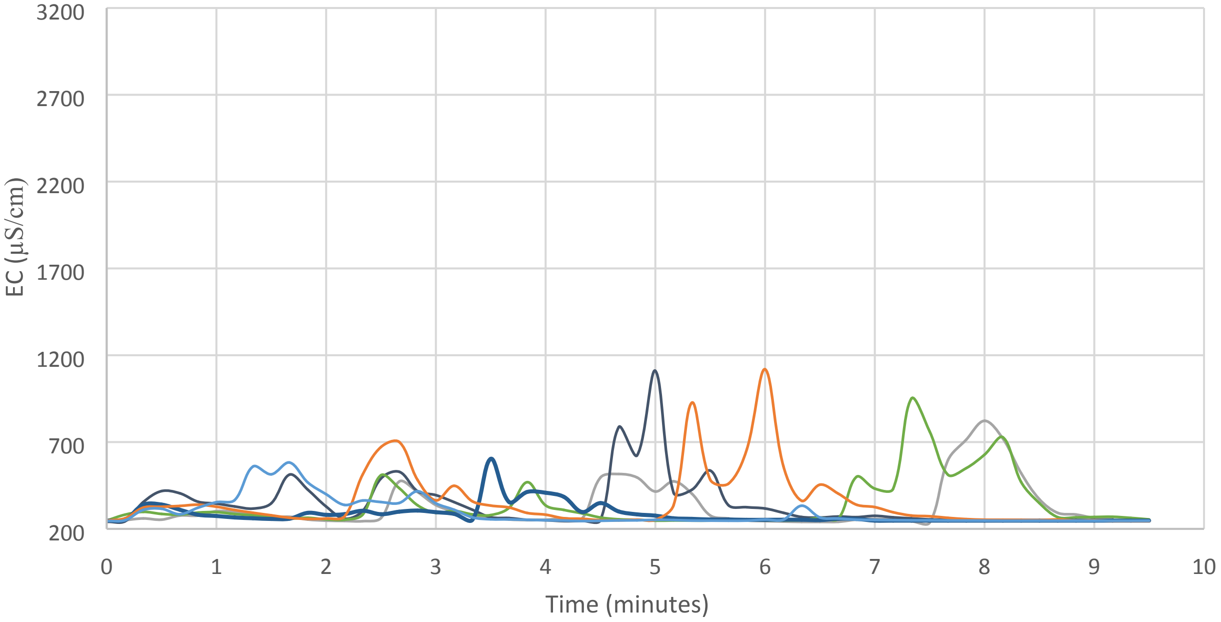


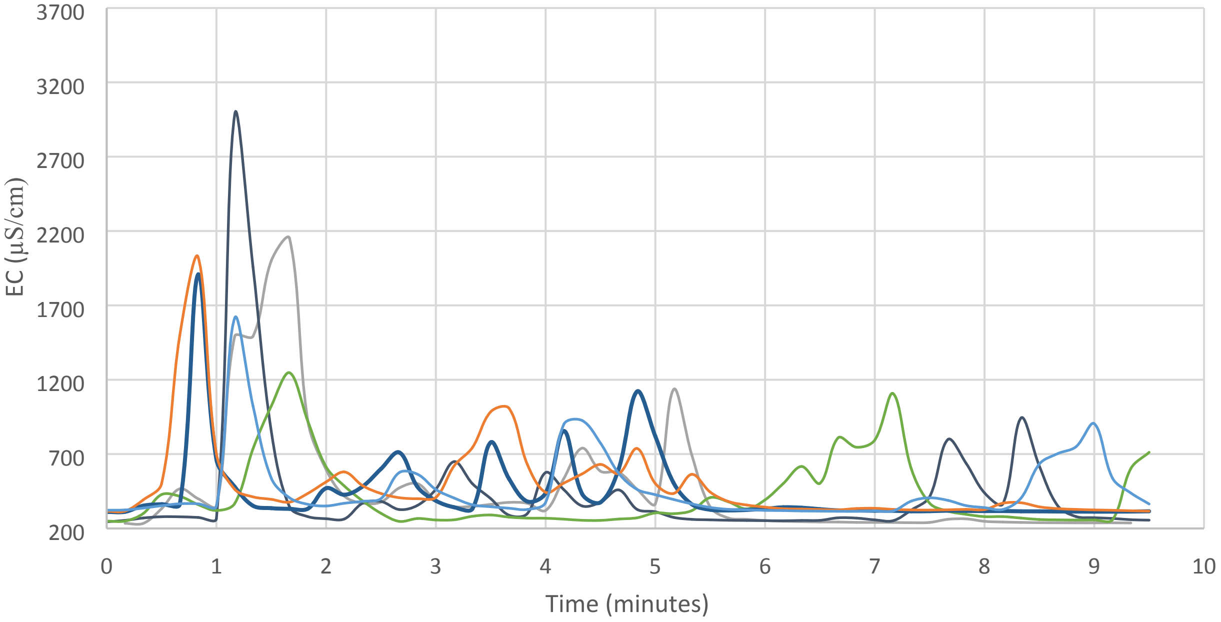
Figure 5. Kinetics data for showers with urine addition. [Conditions: 37.8 L shower volume, 9-10 minute shower time, 25 mL of 25% Campsuds with 0.747 M soda ash, 5 mL of Garnier Fructis biodegradable conditioner, 37°C, 247 μS/cm electrical conductivity treated water]

Based upon comparison of the chemical composition of urine with those of greywater inputs, electrical conductivity (EC) was identified as an inexpensive method of detecting a urination event in the shower in near real time. That is, pure urine may contain ~12,000 mg/L of soluble inorganic salts (40), while the TDS of the soap and conditioner are notably lower, measured gravimetrically at ~7,700 mg/L and ~3,400 mg/L, respectively, and much smaller volumes of soap and conditioner (25 mL and 5 mL, respectively) are added to the system compared with a typical urination. Hence, urine would be expected to increase EC in system water significantly. Therefore, 12 individual test showers were conducted, six of which included a urination event occurring shortly after the shower began, and six did not have a urination event.

Urine monitoring test results are presented in Figure 6. The difference in maximum EC in the drain pipe between showers with urine (1988 ± 585.2 μS/cm) and without urine (489.5 ± 153.6 μS/cm) was determined statistically significant by *T*-test (p = 0.00154). Therefore, EC is suggested as a rapid, inexpensive indication of urination, potentially allowing for an appropriate response. Also, UV intensity dropped rapidly following urination, to a minimum average intensity between the three reactors of 6.50 ± 0.26 mW/cm^2, compared with a minimum average intensity of 7.31 ± 0.31 mW/cm^2 in runs without urination. While a *T*-test reported a p-value of 0.02553, this difference was much less obvious than the difference observed in EC, particularly considering that UV intensity was measured in the reactors where the treatment tank water was recirculating, rather than continuously in the drain pipe. Therefore, there may be a delay between the urine event and detection using UV intensity, and urine-contaminated water will not be as concentrated as in the drain. Other parameters measured did not show significant difference with urination.



(a)



(b)

Figure 6. EC profiles of 12 showers: (a) six that included no urination events and (b) six that included a urination event. [Conditions: 37.84 L shower volume, 9-10 minutes shower time, 25 mL of 25% Campsuds with 0.747 M soda ash, mL of Garnier Fructis biodegradable conditioner, 37^{o}C, 247 µS/cm electrical conductivity in the treated water]

4. Discussion

Overall, advanced oxidation-based net-zero water reuse without biological pretreatment may be practical for a wash station in remote areas, if use is limited to emergency periods and urination is avoided. Alternatively, bromate can be mitigated using a variety of methods, potentially in concert with control of the ozone-UV dosing ratio. Adsorption by GAC is not considered feasible due to limited adsorptive capacity (41), but reduction by GAC may be an option. Bromate reduction with activated carbon can range from 2 mg bromate/g carbon to 94.8 mg bromate/g carbon, depending on water matrix and type of carbon used (42–44). Using a typical ~100 g showerhead filter, this capacity could correspond to monthly replacement, at the flow rate of the pilot plant if reduction sites are destroyed upon reaction. Nitrate may also be addressed by activated carbon, through adsorption (43,45).

Removal of bromide ion in the treatment tank may be a suitable method for mitigating bromate formation (46). In particular, capacitive deionization and selective electrolysis have been shown to remove bromide from the water, advantageous in comparison with a reductive process, in that the accumulation of excess bromide and potential overload of a reduction system can be avoided (41,47). Another option for bromate mitigation is to address bromide sources, including source water, shampoo, and conditioner. In this work, all bromide found in the greywater could be attributed to the soap and conditioner. Therefore, bromate may be mitigated by selection or creation of a bromide-free soap/conditioner, e.g. by military contract. However, further study would be needed to confirm this finding, and bromide might still have to be mitigated in the makeup water stream.

Considering system reuse rate and assuming 18 – 23% RO-treated makeup water with 75% recovery as reported for the US Army SWRS, this system would produce 6-7.7 liters of concentrate per 100 liters of treated water, compared with 33 liters of concentrate per 100 liters of treated water by the SWRS. Regarding system contamination, this system was designed to operate with specific doses of soap/conditioner and avoidance of shock events through detection followed potentially by draining and refilling to maintain water quality. In particular, bromide-free, basic pH, low-TOC soap and conditioner are needed for maximum system efficacy. Equally important, to address the potential system overload of bromide, nitrogen, and TDS associated with urine, an alternative to system drain and refill may be developed. For example, automated flow diversion in response to urine detection through real-time EC measurement may make the system more robust.

In terms of energy consumption, the ozone-UV process has an EEO consistent with literature values and comparable to other AOPs, at an operating EEO of 4.42 kWh/order/m^3 and minimum modeled EEO of 3.73 kWh/order/m^3 (16). This is higher than the energy required for an RO-based system such as the SWRS, which may use around 0.5-3.0 kWh/m^3, depending on source and excluding the pretreatment and concentrate disposal (48). If concentrate must be treated before disposal or source water requires pretreatment, RO costs may increase substantially. In addition, waste produced from this net-zero greywater process is suitable for onsite discharge to replenish the local hydrology, which the makeup water may be drawn from. Finally, this particular system wasted significant energy (400-700 W) by overheating the water and lost this energy to the environment by evaporative cooling. Options for addressing this energy loss may include a storage tank or heat exchanger but must be weighed against potential additional disinfection requirements to address pathogen regrowth.

5. Conclusion and Recommendations

In field implementation, assuming 15-minute shower intervals and 38 L (10 gal) per shower, a capacity of eight showers per treatment batch is projected for the portable advanced oxidation-based NZGW reuse system described in this paper. A subsequent period of additional treatment to achieve <0.5 mg/L TOC is projected to range from 1.45 to 4 hours, depending upon shower spacing and organic loading, with treatment termination signaled by UV_{254} sensor. A portion (15%) of the treated water is recommended for onsite discharge without further treatment, to prevent excessive accumulation of dissolved solids. The following conclusions are drawn:

- The novel fed-batch reactor maintained high UV transmissivity in the reactor, and consistent effluent TOC mineralization, in contrast with a continuous-flow design;
- An advanced oxidation-based net-zero water wash station, with initial charge and makeup water provided by reverse osmosis, provided continuously recycled greywater at an 85% recovery rate for use in a shower with minimal operational requirements, at a production rate of 1,200 L/d (320 gpd) or 32 showers/day, and oxidation capacity of 82 g organics as TOC per day, using soap solution comprising 25% (w/w) Campsuds with 0.747 M soda ash addition and Garnier Fructis biodegradable conditioner, with UV_{254} indicating the end of a treatment period;
- The specific energy consumption of this pilot system is 13.2 kWh/m^3 for UV, 15.9 kWh/m^3 for ozone generation, and 7.93 kWh/m^3 for pumping, resulting in a total energy of 37.0 kWh/m^3 (0.14 kWh/gal), or 1920 W continuous power (3.6 gal diesel fuel/day), while the predicted EEO of a field system with the same ozone:UV dose ratio would be 4.42 $kWh/order/m^3$,

- All US federal drinking water standards were met in the finished water in short-term tests, even with no system drainage and refill, except nitrate and bromate. In addition, if nitrate and bromate standards were adjusted proportionally for a period of up to three years of shower use, these standards would be met as well;
- Total body organics added averaged 1.13 g TOC/shower, with additional amounts (0.64 g male, 2.02 g female) added in the tested soap, shampoo, and conditioner, and electrical conductivity can be used to detect a urination event, which was found to increase organics, nitrogen, and bromide loads to generally unacceptable levels due in part to long urea treatment time;
- Viral inactivation of 7 $\log_{10}$ of MS2 and other microorganisms in a single UV-ozone reactor pass was demonstrated experimentally, and >17.6 $\log_{10}$ inactivation of MS2, >10.6 $\log_{10}$ inactivation of total coliforms, and >14.0 $\log_{10}$ inactivation of *C. parvum* was projected for the proposed fed-batch system.

The following recommendations are made:

- Military field tests of an advanced oxidation-based NZW wash station for temporary emergency deployment;
- Further research into disinfection capabilities and reliability of the process, particularly the testing of each inactivation process separately to verify actual $\log_{10}$ inactivation and compare with calculations;
- Assessment of alternative soaps, and compounds that may be in them, such as triclosan, UV-filters, and emulsifiers;
- Male-female urinal equipment should be integral to the system, with provision for system drain-and-fill in case of urination to the shower drain;

- Additional study of nitrate mitigation methods, particularly activated carbon, so that drinking water standards for long-term use can be met with this system;
- Further research into methods of mitigating bromate once formed, or bromide before bromate formation, are required to meet potable standards, particularly emerging chemical-free electrochemical and experimental methods; and
- Further study of the fed batch system design for UV-ozone mineralization of total organics.

Further research regarding specific organics and other constituents may also be useful.

Conflicts of Interest

There are no conflicts to declare.

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The views expressed in this article are those of the authors and do not necessarily represent the views or policies of the U.S. Environmental Protection Agency. Mention of trade names, products, or services does not convey, and should not be interpreted as conveying, official EPA approval, endorsement or recommendation.

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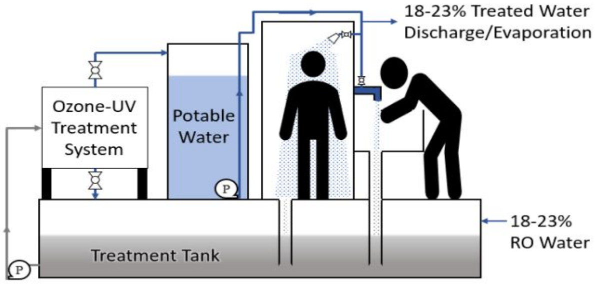

A pilot ozone-UV net-zero greywater wash station was designed, built, and tested, providing a foundation for human contact greywater reuse.