

PEROXIDE AND OZONE: A NEW CHOICE FOR WATER RECLAMATION AND POTABLE REUSE

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Introduction

The use of hydrogen peroxide (peroxide) and ozone together is an advanced oxidation process commonly referred to as peroxone. The combination of ozone and peroxide results in the formation of hydroxyl radicals, which are powerful oxidants. Summarized in Table 1 are the electron oxidation potentials (EOP) of a variety of oxidants used in water and wastewater treatment. In general, the higher the EOP, the more effective the oxidant. Table 1 indicates that hydroxyl radicals have the highest EOP of commonly used oxidants.

Table 1. Electron Oxidizing Potentials (EOP) of Various Oxidizing Agents

| Oxidizing Agent | EOP, V | EOP Relative to Chlorine |
|------------------------|---------------|---------------------------------|
| Hydroxyl radical | 2.80 | 2.05 |
| Ozone | 2.08 | 1.52 |
| Peracetic Acid | 1.81 | 1.33 |
| Hydrogen Peroxide | 1.78 | 1.30 |
| Hypochlorite | 1.49 | 1.10 |
| Chlorine | 1.36 | 1.00 |
| Chlorine Dioxide | 1.27 | 0.93 |

Adapted from *Wastewater Engineering Treatment and Reuse*, Metcalf & Eddy (2003), peracetic acid data courtesy of Enviro-Tech Chemical Services Inc.

Peroxone has been proven for the destruction of recalcitrant micro contaminants such as 1,4 Dioxane, Methyl tertiary-butyl ether (MTBE), Trichloroethylene (TCE), Perchloroethylene (PCE), N-nitrosodimethylamine (NDMA), and other micro contaminants. The same oxidizing potential that makes peroxone useful for these applications, also makes peroxone a powerful disinfectant.

Historically however, peroxone has not been considered for disinfection applications because there is typically little to no residual from which concentration-time (CT) credits can be calculated. The relatively recent acceptance of another 'no residual' disinfectant, ultraviolet light (UV) disinfection, may open the door to other 'no residual' disinfection technologies such as peroxone.

Water Reclamation

A primary concern in water reclamation is disinfection, with potential secondary concerns of disinfection by-product (DBP) minimization and micro contaminant destruction. Potable water reuse

projects go a step further, requiring disinfection, taste and odor removal, micro contaminant destruction, and DBP destruction/minimization. California regulations currently require that potable reuse projects employ reverse osmosis followed by an advanced oxidation process in order to meet combined disinfection/micro contaminant destruction needs.

To date, UV in conjunction with peroxide has been the primary choice for advanced oxidation. The UV/peroxide process, while highly effective, is costly and without competition from other advanced oxidation processes. Peroxone treatment however, may be applicable for such applications. While there is limited information available, it appears that peroxone may be an effective disinfectant. In addition, peroxone provides the additional benefit of micro contaminant destruction, and potential benefits of taste and odor reduction and DBP minimization, all at a competitive cost.

The primary goal of the research presented in this paper was to gain an understanding of peroxone disinfection efficacy and to determine the optimum ozone dose and peroxide/ozone molar ratios required to obtain target virus disinfection goals. The data presented in this report are based on the results of testing conducted on a proprietary peroxide/ozone advanced oxidation system manufactured by Applied Process Technology, Inc. (APPLIED), termed HiPOx.

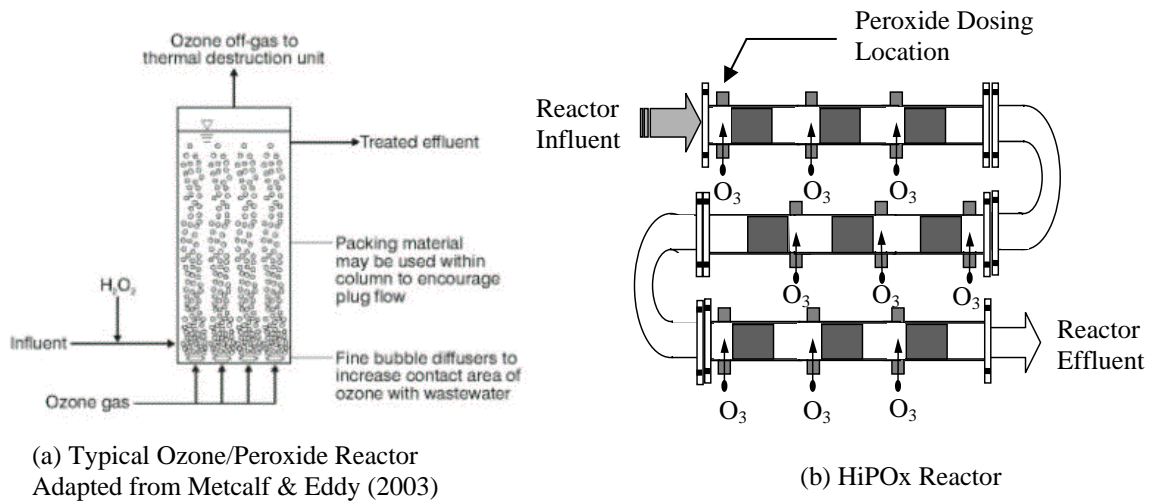
Results to date indicate that using ozone in conjunction with peroxide is an effective disinfectant. Using ozone doses and peroxide/ozone ratios of 0.7 mg/L and 0.5 respectively, resulted in greater than 7-log inactivation of MS2 with a contact time of approximately 100 seconds. This is on the order of what one would expect using ozone alone, but there is a potential benefit to peroxide/ozone over ozone alone, micro contaminant reduction and DBP minimization.

HiPOx Advanced Oxidation System

HiPOx is a proprietary advanced oxidation process employing a variation on peroxone treatment. HiPOx is manufactured and distributed by APPLIED of Pleasant Hill, California. The HiPOx system was developed, and is currently marketed, as a tool for destroying a wide variety of recalcitrant water contaminants, including petroleum constituents MTBE, t-butyl alcohol (TBA), benzene, toluene, ethylbenzene, and xylene (collectively referred to as BTEX), NDMA, and the solvent stabilizer 1,4 dioxane. The ability of the HiPOx system to remove such recalcitrant contaminants has been successfully proven, though the data are not presented in this paper.

The difference between typical peroxone treatment and HiPOx treatment is primarily in the reactor hydraulics and the chemical injection locations. In most cases, ozone is added at a single point at the head of an open ozone contact chamber. Peroxide is added at a location downstream of the ozone injection point. In contrast, the HiPOx reactor is a pressurized in-vessel system, in which peroxide is dosed first. The ozone dose is equally distributed over up to 18 injection points throughout the reactor (Figure 1).

The work presented in this report is intended to provide a preliminary evaluation of the HiPOx system for disinfection, sufficient to allow for comparisons to other conventionally used disinfection technologies. A limited discussion of the ability of the HiPOx system to minimize standard disinfection byproducts (DBPs) is also presented.

Figure 1. Peroxone Reactor Schematics (a) Typical Reactor (b) HiPOx Reactor

Literature Review

Prior to the start of field research on peroxone disinfection for this project, Carollo was tasked to perform a literature search on the use of peroxone for disinfection and DBP minimization. The results of the literature review are presented below.

Disinfection

Limited studies have investigated the impact of peroxide addition on the efficacy of disinfection with ozone. Overall, it appears that peroxide addition decreases the effectiveness of ozone as a disinfectant for a given applied ozone dose. However, the decrease does not appear to be severe until very high hydrogen peroxide to ozone ratios (e.g., >0.5) are reached.¹

In some cases, it has been concluded that peroxone (ozone and hydrogen peroxide) is equally or more effective than ozone for pathogen inactivation. Such statements must be read with caution however, as the comparison may be based on the ozone residual, rather than the applied ozone concentration. As peroxide greatly decreases the ozone residual, a much greater applied ozone dose would be required to achieve the same residual.

There are three articles of particular interest that are based on work conducted by the Metropolitan Water District (MWD) in Southern California.

One study investigated four ozone doses (0.5, 1.0, 2.0, 4.0 milligrams per liter [mg/L]) and four peroxide to ozone ratios (0, 0.3, 0.5, 0.8). Testing was conducted on two waters: Colorado River Water

¹ All ratios of peroxide to ozone are expressed in this report as mass ratios unless otherwise noted.

(CRW) and State Project Water (SPW). Compared to CRW, SPW has lower alkalinity, higher TOC, and higher ozone demand. The results of the study for *E. coli* and MS2 bacteriophage on SPW and CRW indicate that microorganism disinfection by peroxone may be very dependent on both the peroxide/ozone ratio and on source water quality (Wolfe *et al.*, 1989).

For *E. coli*, the results reasonably demonstrated that increasing peroxide to ozone ratios led to decreased disinfection efficacy. This impact was particularly severe when high doses of ozone were used. For example, in SPW at an applied ozone dose of 4 mg/L, the inactivation of *E. coli* decreased from >7.9 log at a ratio of 0 to 1.9 log at a ratio of 0.8. In CRW the impact was less severe, however a decrease from >7.7 to 5.7 log inactivation was observed for the same ozone dose and ratios.

For MS2, the overall relationship between peroxide to ozone ratio and inactivation efficacy was less clear. The highest peroxide to ozone ratio (0.8) was detrimental to inactivation, as compared to no peroxide addition, at all ozone doses and in both waters. However, the intermediate ratios (0.3 and 0.5) had little impact on disinfection efficacy. Observed small variable increases and decreases in log kills were likely due to experimental variation and are not significant. A second study by Ferguson *et al.* (1990) investigated the impacts of ozone and peroxone on the inactivation of *Giardia muris*, *E. coli*, and the coliphages MS2 and f2. Three ozone doses (1, 2 and 4 mg/L) and four peroxide to ozone ratios (0, 0.1, 0.2, and 0.3) were used, all at a contact time of 12 minutes. The results on *E. coli*, MS2, and f2 were inconclusive, as there was almost complete inactivation in all experiments (seeded concentration was 10^7 to 10^8 pfu/mL and observed inactivation ranged from 6.3 to 8.1 logs). The only conclusion that can be drawn from these results is that any deterioration of the inactivation efficacy of ozone due to peroxide addition was not significant enough to prevent near-complete inactivation.

Giardia testing was limited to two ozone doses (1.0 and 2.0 mg/L) and two peroxide to ozone ratios (0, 0.2). The peroxide addition was found to have little effect on inactivation.

Wolfe *et al.* (1989b) discussed the work done at MWD, as described by Ferguson *et al.* (1990), and presents many of the same results. However, one additional finding is that the inactivation of *Giardia* by peroxone was dependent on contact time (6 versus 12 minutes), whereas inactivation of viruses and bacteria was not. *This finding is potentially highly significant for the application of peroxone to wastewater treatment and water reclamation, as detailed further on in this document.* Wolfe determined that for a 2-log reduction of *Giardia* and an applied ozone dose of 1.0 mg/L, the contact time required for ozone alone (8 minutes) was slightly lower than that required for ozone with peroxide added at a ratio of 0.2 (9 minutes).

Disinfection Byproduct Minimization

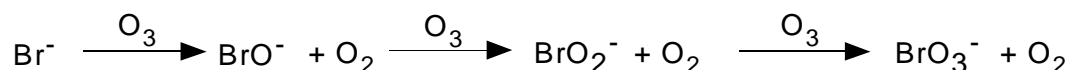
The most important DBP produced by ozonation processes is bromate. Bromate is currently regulated in drinking water at a maximum contaminant level (MCL) of 10 µg/L by the EPA Stage 1 Disinfectants/DBPs (D/DBP) Rule. Bromate is not a priority pollutant. However, it is regulated for facilities practicing indirect potable reuse.

As with disinfection, there is some confusion in the literature when discussing the impact of peroxide addition on bromate formation, as specific studies may be investigating one of two situations: (1) application of peroxide with additional ozone such that the ozone residual remains equivalent (referred to as constant residual) and (2) application of peroxide with the applied ozone dose, and a lower ozone residual (referred to as constant dose). In general, the former case will lead to greater bromate formation

with peroxide addition, due to the greater ozone dose that must be applied to maintain the residual. The latter case generally leads to a decrease in bromate formation.

The formation of bromate can occur by either of two mechanisms: Molecular Ozone Mechanism (Glaze et al 1993, Haag and Hoinge 1983, and Von Guten et al 1995) or Radical Mechanism.

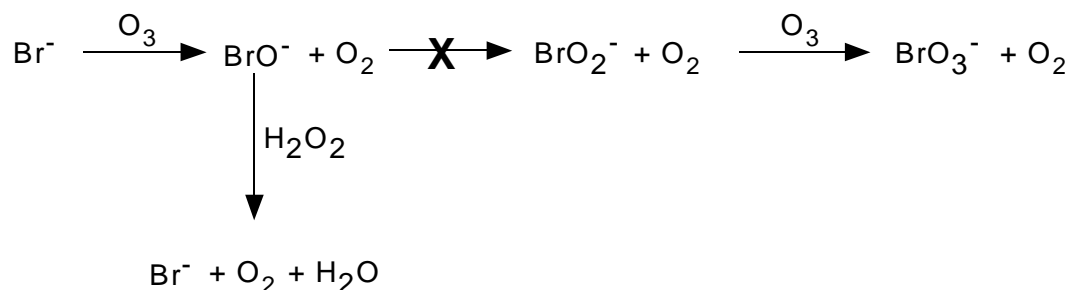
Molecular Ozone Mechanism:



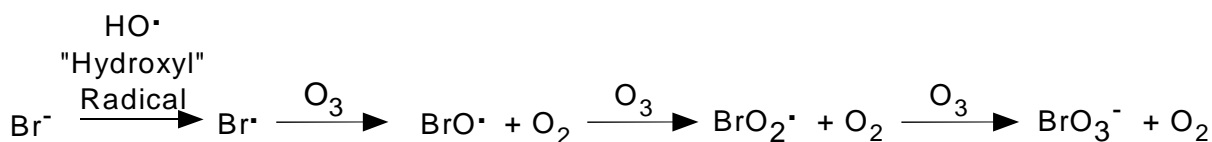
Equation 1

In the Molecular Ozone Mechanism the bromide is first oxidized to hypobromite (-OBr) by ozone. The hypobromite ion is then further oxidized by ozone to bromate. The addition of hydrogen peroxide stops the oxidation of hypobromite by ozone (Von Guten and Oliveras 1997). The hydrogen peroxide rapidly converts the hypobromite back to bromide.

The Radical Mechanism:



Equation 2



Equation 3

In the Radical Mechanism the bromide is first converted to a bromide radical by hydroxyl radical (HO•) (Wasterhoff et al. 1994). The bromide radical reacts with molecular ozone to yield hypobromite radical (Von Guten and Oliveras 1998). The hypobromite radical is then further oxidized to bromate. In the radical mechanism, the key intermediate is the hypobromite radical. Unlike hypobromite ion in the Molecular Ozone Mechanism, the hypobromite radical does not react with hydrogen peroxide to yield bromide.

In bromide containing water, both the Molecular Ozone Mechanism and the Radical Mechanism are pathways to bromate. Bromate formation is minimized by controlling the concentration of ozone available.

Von Gunten et al. (1996) presented an excellent review of the impact of peroxide addition on bromate formation during ozonation. The authors note that peroxide addition for a constant applied ozone dose (constant dose process) influences bromate formation through two mechanisms: (1) peroxide increases production of hydroxyl radical, increasing bromate formation, and (2) peroxide increases ozone decay leading to a lower ozone residual, which decreases bromate formation. The former mechanism tends to dominate during initial contact, leading to increased bromate formation for very short contact times, whereas the latter process dominates at longer retention times leading to overall lower bromate formation in reactors with longer retention times. A greater peroxide dose will exacerbate both mechanisms, leading to greater bromate formation initially, but typically lower effluent bromate levels.

In the HiPOx system bromate control is achieved in two main ways. As shown in the discussion below, the addition of hydrogen peroxide stops the oxidation of the hypobromite intermediate by ozone (Von Guten and Oliveras 1997). Hydrogen peroxide converts the hypobromite ion back to bromide. The use of excess hydrogen peroxide will control the formation of bromate. The second way in which the HiPOx system may minimize bromate formation is related to hydraulic efficiency and chemical injection locations unique to the HiPOx system. The hydraulic efficiency of the reactor along with the multiple smaller doses of ozone throughout the reactor should result in better chemical mixing. The fact that ozone is added after peroxide and because the reaction of peroxide with ozone is very rapid, it is expected that ozone would only be available to react with bromide for a very short time. Both of these conditions may limit the bromate formation.

Experimental Methods

The research presented in this report was intended only to provide information on the disinfection performance of the HiPOx system. As a part of this, the reactor hydraulics must also be evaluated since the hydraulics of the HiPOx reactor are expected to be very different from the hydraulics found in typical ozone contactors. This preliminary evaluation consisted of testing only one flow rate through the system, 10 gallons per minute (gpm).

The test results research presented in this report were conducted on the settled water at a water treatment plant in California. As the settled water is of high quality, disinfection experiments required seeding of MS2 coliphage, a common surrogate for determining disinfection efficacy. The waste from the system was sent directly to a contracted disposal tank that provided double-containment.

Hydraulic Characterization

The first step in evaluating the disinfection performance is to understand the hydraulic efficiency of the HiPOx reactor. This was accomplished by conducting a tracer test on the system using a conservative tracer. The volume of the pilot-scale reactor is 16.3 gallons. At 10 gpm, the theoretical detention time (T) is 98 seconds. The tracer test was designed to determine the time required for the influent and effluent to reach equilibrium, as well as to determine the t_{50} (the modal contact time for the reactor) for the system. The t_{50} is of importance due to its standard use in the calculation of chlorine contactor disinfection credit for water reclamation applications. To determine t_{50} , a step tracer test was conducted. As a system approaches plug flow, the t_{50}/T will approach 1.

The tracer study was conducted using instant coffee as the conservative tracer. Ultraviolet transmittance (UVT) measurements, providing a direct correlation to instant coffee concentrations, were used to evaluate the reactor hydraulics. The tracer test was conducted once on each of two consecutive days. For the first test, coffee was dosed at a concentration that resulted in a steady-state influent UVT of 11 percent. The second test was conducted at a concentration resulting in a steady-state influent UVT of 83 percent. The first test was conducted to give the most detail in the curve as the effluent UVT declines. The second test was conducted to ensure that the UVT meter had not reached its detection limit at 11 percent and that the system had, in fact, reached equilibrium.

Disinfection

The preliminary evaluation of the disinfection capabilities of the HiPOx system involved adding a concentrated virus (MS2) to the pilot system influent. The concentration of the MS2 stock obtained from BioVir Laboratories was approximately 10^{11} plaque forming units (pfu)/mL. The influent MS2 concentration goal was on the order of 10^7 pfu/mL.

To obtain an estimate of the disinfection capabilities of the HiPOx system, four disinfection tests were initially conducted. For each of the exploratory tests, only one influent and one effluent sample were collected. Table 2 shows the dose schedule used for the exploratory disinfection tests. The results of these tests indicated that the ozone doses were too high, as complete inactivation of the seeded MS2 virus was achieved.

Due to this finding, a second round of exploratory tests were performed, at ozone doses of 0.25 and 0.5 mg/L. The test scenarios for the second round of exploratory testing are also shown in Table 2. The results of the second round of exploratory testing were used to determine the dosing strategies for the preliminary evaluation of the HiPOx system’s disinfection capabilities. The dosing strategies for the disinfection tests are located in Table 3. Each test was performed twice. One influent and three effluent samples were collected for each of the tests.

Table 2. Exploratory Disinfection Tests

| Test ID | Ozone Dose, mg/L | Peroxide/Ozone Mass Ratio |
|------------------|------------------|---------------------------|
| A ⁽¹⁾ | 1 | 0.5 |
| B | 2 | 0.5 |
| C | 1 | 0.5 |
| D | 1 | 0 |
| F ⁽²⁾ | 0.25 | 0.25 |
| G ⁽²⁾ | 0.25 | 0.5 |
| H ⁽²⁾ | 0.25 | 0 |
| I ⁽²⁾ | 0.5 | 0 |

Notes:

(1) Test A was discarded due to high concentration of chlorine found in the seed mixture. The source water for the remainder of the tests was changed to a source with no measurable chlorine concentration.

(2) These tests were performed as part of the second round of exploratory disinfection tests.

Table 3. Preliminary Evaluation of MS2 Disinfection

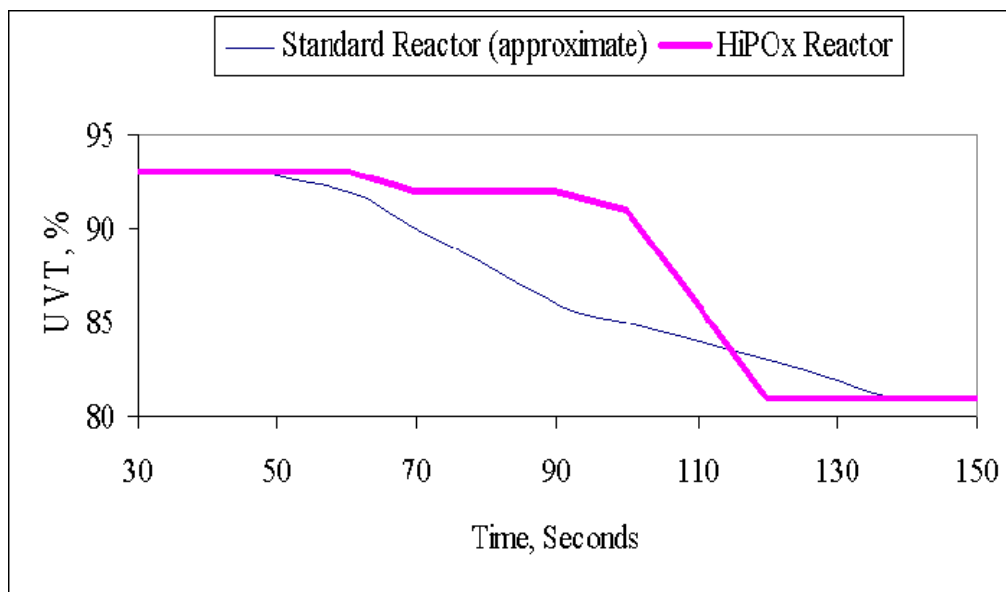
| Test IDs | Ozone Dose, mg/L | Peroxide/Ozone mass ratio |
|----------|------------------|---------------------------|
| J, AA | 0.3 | 0.00 |
| K, Z | 0.7 | 0.00 |
| L, Y | 1 | 0.00 |
| M, X | 0.3 | 0.76 |
| N, W | 0.7 | 0.38 |
| O, V | 1 | 0.25 |
| P, U | 0.3 | 1.52 |
| Q, S | 0.7 | 0.75 |
| R, T | 1 | 0.50 |

Test E was a quality control test designed to show that there is no inherent loss of MS2 in the HiPOx reactor. For this test, the same influent concentration of MS2 was passed through the system as with the other tests, but no peroxide or ozone was dosed.

Experimental Results

Hydraulic Characterization

Tracer tests were used to characterize the hydraulics of the pilot-scale HiPOx system. Two tracer tests were conducted and UVT was the measured parameter. The resulting t_{50} was 98 seconds for both tests. Figure 2 shows the results of one of the tracer tests as well as hypothetical data of a typical ozone contactor to illustrate the difference in reactor efficiency. Using a t_{50} of 98 seconds, and a theoretical contact time (T) of 98 seconds as previously calculated, the efficiency of this reactor is 100 percent (or plug flow), which far exceeds the anticipated efficiencies for standard ozone reactors (~50 percent to 80 percent efficiency).

Figure 2. HiPOx Tracer Test Results Compared to Typical Ozone Reactor

Disinfection

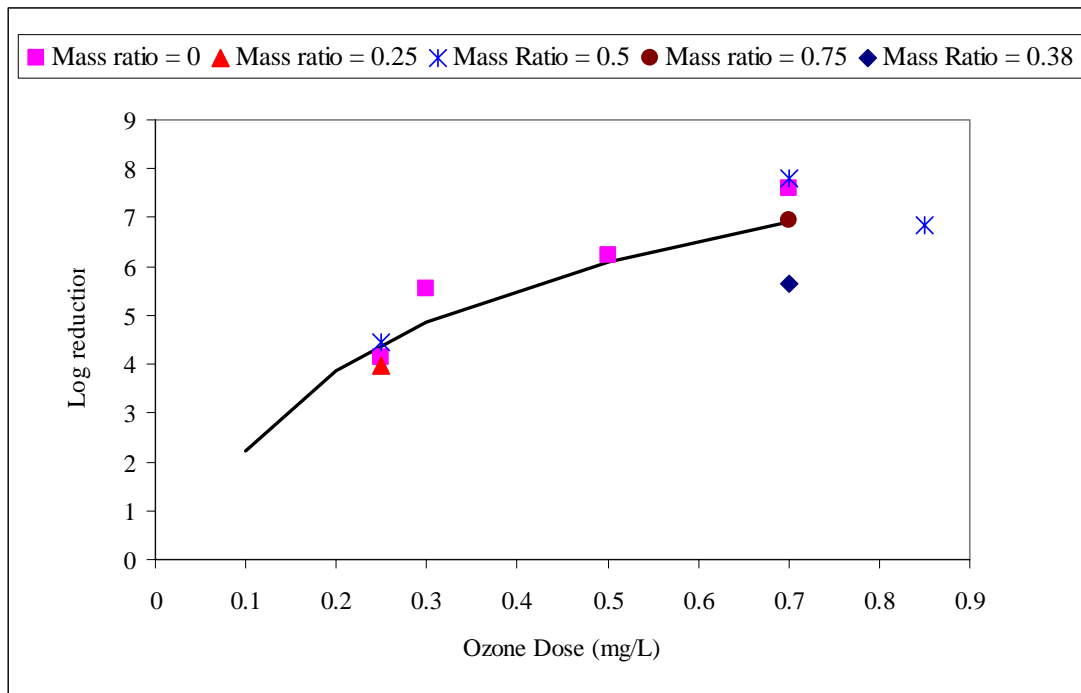
Initial disinfection testing conducted on the HiPOx system was conducted at ozone doses of 1 and 2 mg/L. These ozone doses proved to be too high, as almost complete inactivation of the seeded MS2 virus was achieved. Additional testing was conducted in which the ozone dose was reduced to 0.25 and 0.5 mg/L, with different peroxide/ozone mass ratios. These tests showed promising results so final testing was conducted in accordance with the schedule listed in Table 3.

Table 4 summarizes the results of the tests in which complete inactivation did not occur. Two independent variables, ozone dose and peroxide/ozone mass ratio, were used for performing a log linear regression of the data. The results of this regression analysis are shown in Figure 3 along with the actual data points obtained for each test.

Table 4. Preliminary Evaluation of MS2 Disinfection

| Test IDs | Ozone Dose, mg/L | Peroxide/Ozone mass ratio | Log MS2 Reduction |
|----------|------------------|---------------------------|-------------------|
| A | 0.9 | 0.50 | 6.85 |
| C | 0.7 | 0.5 | 7.81 |
| F | 0.25 | 0.25 | 3.96 |
| G | 0.25 | 0.50 | 4.46 |
| H | 0.25 | 0 | 4.14 |
| I | 0.5 | 0 | 6.22 |
| J | 0.3 | 0.75 | 6.96 |
| K | 0.7 | 0 | 7.61 |
| N | 0.7 | 0.38 | 5.63 |

Figure 3. Disinfection of MS2 Coliphage



At a minimum, these results support what is already known, that ozone is a very effective disinfectant. At doses of less than 1 mg/L, up to 7-log inactivation of MS2 was observed. As the results are limited, it is not possible to quantify the impact of peroxide addition on disinfection efficacy – individual results appear to indicate small positive or negative effects. Most significantly, peroxide addition did not appear to greatly decrease the effectiveness of ozone (any impacts appear to be small).

Market Applications

The HiPOx system has a potential market in water reuse applications, both landscape irrigation and indirect potable reuse. For both of these applications, the performance target for HiPOx is disinfection of bacteria and virus. The requirements for recycled water disinfection vary from one State to another. The current major markets for landscape irrigation with recycled water are California, Arizona, and Florida, Oregon, Nevada, Texas, and Washington. Each of these states has bacterial disinfection requirements. California has a standard of 2.2 most probable number (MPN) for total coliform per 100 mL, while some other states have non-detect fecal coliform requirements per 100 mL. California is the only state to specify the 5-log inactivation of virus for all non-chlorine disinfection technologies. In addition, only California is currently requiring treatment beyond disinfection for indirect potable reuse projects (some states require nitrogen removal), with Draft Groundwater Recharge Regulations specifying advanced oxidation for micro contaminant destruction as well as total coliform and virus disinfection. Based upon the research performed in this project and preliminary cost comparisons to other conventionally used technologies, the HiPOx system appears to be a cost competitive water reuse treatment option as either an ozone only system or as a peroxone system. However, further confirmation of the efficacy of the technology is needed.

Water Reuse for Landscape Irrigation

In California, water reuse for landscape irrigation generally falls under the “tertiary recycled water” requirements of Title 22 of the California Code of Regulations. Chlorine disinfection (either free or combined), preceded by media filtration, is the most common method of disinfection for this application. The frequent alternative to chlorine disinfection for “tertiary recycled water” disinfection is UV, also preceded by media filtration. Recent manufacturing efficiencies have resulted in the more frequent employment of membranes (in this case microfilters) in lieu of media filtration. Utilization of the HiPOx system, in ozone only mode, or in the peroxone mode may be an efficient alternative to post filtration chlorination or UV disinfection.

Additionally, the HiPOx process, with rigorous testing, may provide the ability to produce recycled water quality effluent without filtration (thus potentially becoming highly cost-effective). The possibility of being able to meet reclaimed water regulations without filtration is unique to the HiPOx system. While it would be possible to achieve the necessary disinfection with chlorine disinfection alone, filtration serves to remove organic matter. Without filtration chlorine disinfection would likely result in the formation of high concentrations of disinfection by products. Filtration is required prior to UV disinfection to remove particles that can potentially shield microorganisms from the UV light.

Preliminary results indicate that HiPOx may be more cost effective than other disinfection technologies. However, additional confirmation is necessary. An additional selling point for APPLIED is the ability of

HiPOx to also destroy micro contaminants, which is not as easily achieved for chlorine or UV at disinfection levels.²

Water Reuse for Indirect Potable Reuse

The current “state-of-the-art” for new indirect potable reuse projects in California and elsewhere (primarily Southern California) is the employment of microfiltration (MF), reverse osmosis (RO), and advanced oxidation for the repurification of conventionally treated wastewater. Chlorine disinfection is not the current method of choice for post-RO disinfection, as it creates unwanted DBPs, primarily NDMA. The now-standard post-RO disinfection method is UV, with hydrogen peroxide dosing capabilities available for micro contaminant destruction (most notably NDMA and 1,4-dioxane). The potential does exist for the application of HiPOx followed in series by UV. This may provide multiple benefits, as HiPOx could fully oxidize compounds such as 1,4 Dioxane and partially oxidize NDMA, while UV could provide a measure of disinfection and complete the destruction of NDMA to below the regulated level (10 ppt action level). The potential also exists for HiPOx to be the stand-alone disinfection/advanced oxidation solution for post RO effluent.

Recommended Future Work

Of critical importance to the application of HiPOx for water reuse (especially in California and Florida) is the demonstration of disinfection performance. In California, such testing would be properly preceded by the submittal and approval of a testing protocol to the California Department of Health Services (CDHS). The testing would include the demonstration of meeting the 2.2 MPN/100 mL total coliform level and meeting the 5-log reduction of poliovirus (or demonstrating the reduction of MS2 that correlates to 5-log reduction in poliovirus). The testing would also include the rigorous testing and correlation of disinfection dose and performance to aggregate wastewater parameters such as BOD, TSS, COD, TOC, and turbidity. If source water quality is found to have a significant effect on disinfection efficacy, a process should be developed for determining the optimum ratio for individual clients. The results from such robust testing must be submitted to CDHS and must be approved prior to the use of HiPOx in California for water reuse. This process for protocol development and testing is in the planning stages and has yet to be implemented.

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² Some Regional Water Quality Control Boards in California are implementing Action Levels (ALs) for tertiary recycled water permits. These ALs include some organics at low concentrations.

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