Advanced Oxidation Technology

Introduction
Developments in chemical water treatment have made several oxidative technologies feasible. These technologies are based on the generation of highly reactive intermediates that initiate a sequence of reactions resulting in the destruction and removal of organic pollutants are generally referred to as Advanced Oxidation Processes (AOTs). All AOTs are based mainly on hydroxyl radical (HO\textsuperscript{•}) chemistry, which is major reactive intermediate responsible for organic substrate oxidation. In our system, we will use ozone and hydrogen peroxide. Ozone is a very reactive compound that has the ability to chemically attack many organic compounds, initiating their oxidation. Ozone is typically used in conjunction with hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}) to speed up the reaction, since H\textsubscript{2}O\textsubscript{2} breaks down to produce HO\textsuperscript{•} radicals and gets reactions going faster. For more info go to:

Laboratory setup
Ozone is generated in an ozone generator by sending an electric charge through oxygen (O\textsubscript{2}). The O\textsubscript{3}/O\textsubscript{2} mixture is delivered through a microporous stainless steal tube that produce small gas bubbles, which are injected into water contaminated with organic pollutants. The dissolved ozone then reacts with the organic pollutants and breaks them down into small molecules. The laboratory setup is shown in Figure 1.

Figure-1 Schematic representation of the experimental setup
Laboratory procedure

- Make sure that you have safety equipment (glasses, gloves, etc.) and are familiar with safety devices (eye wash, shower, etc.)
- Become familiar with all the components before starting.
- Determine the operating conditions for your experiments before you turn anything on. Make sure you decide on the frequency for taking samples, sample size and container, data needed, etc.
- Samples to be used to determine the initial toluene and MTBE concentrations can be collected at the inlet of the reactor before turning on the ozone. I recommend collecting 2-3 samples so that you can do some variation analysis later on. Collect samples into 100 ml beakers and immediately transfer into 40 ml amber or clear vials with clean caps and septa.
- Label all your sample vials before you start.
- The vials must be completely filled with liquid to prevent volatilization of organics into headspace, which will affect your results.
- When you are finished collecting the initial samples, turn on the ozone.
- To start up the Ozone generator:

  1. Turn on O3 sensor (portable) and check battery;
  2. Turn on the O2 cylinder connected to the O3 generator and make sure there is enough O2 left in the cylinder (at least 500 psi for one experiment);
  3. Plug the O3 destructor and make sure it gets warm before your turn on the O3 generator (at least 10 minutes);
  4. Turn on O3 analyzer and wait until reading is equal or close to zero; \textbf{\textit{until the ozone analyzer is back from repair, we are skipping this point. We will use the set points in the ozone generator}}
  5. Turn on cooling water for the O3 generator and check for water flow behind O3 generator, draining to the small sink;
  6. Open **Valve 1** all the way (counter clockwise);
  7. Turn on O3 generator by switching **Mains** on, then push the **PSU** switch on to start the flow of O2;
  8. Open **Valve 3** (near the bottom of the glass reactor) to let the air flow into the glass reactor; you might be able to see small gas bubbles in the reactor;
  9. Reduce oxygen flow with **valve 1** so rotometer is at 25 at top of silver ball (1000 ml/min);
  10. Adjust flow rate to 3.0-3.5 (at the top of the red cone) by using **Hand Control Valve 201** on the O3 generator (pressure indicator on O3 generator should be kept at about 20 psi);
  11. Adjust **Valve 2** to have a flow rate of 500 ml/min through the O3 analyzer; \textbf{\textit{(Since we don’t have the ozone analyzer for the experiment, we do not need to adjust valve 2)}}
  12. Allow about 2-3 minutes for all the conditions to stabilize and make adjustments to get the right flowrates;
(13) Press “Set Point Up” button (on O₃ generator) to generate O₃; for each 5% increase, you must wait at least 15 seconds;
(14) Increase the ozone concentration as needed by pressing the “Set Point Up” button;

- Turn on the water pump and adjust Valve 4 to obtain about 1.0 L/min.
- Each team will test two different O₃/H₂O₂ combinations. Turn on the H₂O₂ pump and run it at 0, 10, 15, 20, 25 or 35 mL/min.
- Run each condition for at least 3 minutes before taking samples, to allow the system to reach steady state (not equilibrium).
- Take 4 samples every 3 minutes. (2 at the inflow and 2 at the outflow). Use the same procedure as before. You should keep track of the time since the beginning of operations, and note any discrepancies.
- Label samples with the location of sample taken, the O₃ concentration and the sample number.
- Once you are finished with your two test conditions, you need to turn off the system:

  1. Press “Set Point Down” (on O₃ generator) to 0% (no need to wait 15 seconds);
  2. Turn on Valve 1 all the way;
  3. Turn off Valve 3 (make sure no water comes in);
  4. Switch PSU off, let O₃ generator automatically purge to get rid of the O₃ in the generator (100 minutes);
  5. Turn off the Mains switch;
  6. Turn off cooling water;
  7. Turn off O₃ analyzer;
  8. Turn off water pump;
  9. Unplug O₃ destructor;
  10. Make sure all lines are dry;
  11. Turn off O₂ gas;
  12. Turn off O₃ sensor.

- We will use GC/MS to analyze all of your samples, so transfer 1.5 ml of each of your samples to EPA 1.5 ml vials. Be sure to label each vial before you bring them to Peng.
- Present your results in terms of removal efficiency as a function of O₃/H₂O₂ combination. Note the differences between MTBE and toluene. What are the key physicochemical properties and conditions for this process?

The following information is provided for your reference:
- 1 SCFM = 1 standard cubic feet per minute = 0.472 L/s = 28.32 L/min
- 1 bar = 14.5 lbs/square inch (psi) = 1 bar = 10⁵ Pascal = 10⁵ N/m²
- 1 gallon = 3.7854 L