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Copper-Catalyzed Peroxide Oxidation Testing for Tetraphenylborate Decomposition

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Abstract: A new processing option, copper-catalyzed hydrogen peroxide oxidation of tetraphenylborate under alkaline conditions, was demonstrated in laboratory testing. Laboratory-scale tests were conducted to evaluate the use of copper-catalyzed hydrogen peroxide oxidation to treat simulants of the Savannah River Site tank waste. The oxidation process involves the reaction of hydrogen peroxide with a copper catalyst to form hydroxyl free radicals. With an oxidation potential of 2.8 volts, the hydroxyl free radical is a very powerful oxidant, second only to fluorine, and will react with a wide range of organic molecules. The goal is to oxidize the tetraphenylborate completely to carbon dioxide, with minimal benzene generation. Testing was completed in a lab-scale demonstration apparatus at the Savannah River National Laboratory. Greater than 99.8% tetraphenylborate destruction was achieved in less than three weeks. Offgas benzene analysis by a gas chromatograph demonstrated low benzene generation. Analysis of the resulting slurry demonstrated >82.3% organic carbon destruction. The only carbon compounds detected were formate, oxalate, benzene (vapor), carbonate, p-terphenyl, quaterphenyl, phenol, and phenol 3-dimethylamino.

Keywords: Organic, oxidation, high-level waste

INTRODUCTION AND BACKGROUND

One Savannah River Site (SRS) high-level waste (HLW) tank currently holds legacy material containing organic tetraphenylborate compounds from the

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operation of the In-Tank Precipitation process. This material is not compatible with the waste treatment facilities at SRS and must be removed or undergo treatment to destroy the organic compounds before the tank can be returned to service in the tank farm. The HLW tank currently contains ~240,000 gallons of alkaline slurry with potassium and cesium tetraphenylborate solids. The main radioactive component is ^{137}Cs.

A number of research efforts focused on developing processing schemes to decompose tetraphenylborate (TPB). Testing of hydrolysis, catalyzed hydrolysis (1), and steam reforming (2, 3) received funding from the tanks disposition project. The Department of Energy (DOE) Office of Cleanup Technology (EM-21) also directly funded studies on Fenton's chemistry (4–6) for treatment of the waste.

This work extends the work to destroy the organics using Fenton's reagent (7, 8). The research examined copper hydrolysis and peroxide oxidation processing strategies for organic destruction in-tank, but testing failed to meet the destruction efficiency of 2.2–7.7 mg/L/hr equivalent TPB decomposition to achieve TPB decomposition in less than one year.

This testing with non-radioactive simulant determined the rate and extent of tetraphenylborate decomposition as well as the rate and type of potassium tetraphenylborate offgas and liquid decomposition products for the Fenton's reaction chemistry.

Most TPB decomposition processes, such as hydrolysis or catalytic decomposition, form benzene as a byproduct. The potential for benzene production complicates the safe operation of any process for destroying the TPB in the HLW tank. Benzene is flammable at concentrations between 1.2 and 7.8 volume percent in air (9). Because of benzene's volatility, a flammable mixture of benzene and air is easily formed. Benzene is also a carcinogen with an OSHA Permissible Exposure Limit of 1 part per million (10). The combination of potential flammability and toxicity makes controlling benzene a critical parameter to solve. Unresolvable issues resulting from higher than expected benzene production from TPB decomposition led to the eventual shutdown of the In-Tank Precipitation Facility in 1998.

Processing that completely oxidizes TPB to carbon dioxide and water is advantageous because it will minimize or eliminate the benzene hazards as well as organic byproducts. Fenton's reagent, a combination of hydrogen peroxide with typically an iron catalyst at pH 3–5, is used to treat a variety of industrial wastes containing a range of toxic organic compounds (e.g., phenols, formaldehyde, and complex wastes derived from dyestuffs, pesticides, wood preservatives, plastics additives, and rubber chemicals). The process has been applied to wastewaters, sludges, and contaminated soils (11). Use of Fenton's Reagent has solved a number of environmental problems involving the destruction of organic contaminants in soil and groundwater (12). However, Fenton's Reagent requires a pH of 3–5 for optimal decomposition. Since a reaction at pH 3–5 would be too corrosive for a carbon steel waste tank, the combination of the right catalyst with hydrogen peroxide at a pH above 10.3 is desirable.

The overall reactions for the tetraphenylborate ion and phenol are shown in equations (2) and (3). One of the prime reasons H_2O_2 is advantageous is that it leads to benign reaction products (i.e., water and carbon dioxide). Based on these proposed reactions, 60 moles of H_2O_2 are required per mole of tetraphenylborate and 14 moles of H_2O_2 are required per mole of phenol. As a result, 2.07 moles of H_2O_2 are required to destroy the TPB and 0.085 moles of H_2O_2 is required to destroy phenol (for a total of 125 mL of peroxide) for 0.600 L of slurry. Note that excess peroxide is generally required for efficient reaction rates.

$$B(C_6H_5)_4^- + 60H_2O_2 \longrightarrow 24CO_2 + 70H_2O + H_2BO_3^- \qquad (1)$$

$$C_6H_5OH + 14H_2O_2 \longrightarrow 6CO_2 + 17H_2O \qquad (2)$$

Two experiments were completed to determine the TPB degradation under proposed processing conditions. All the experiments used a simulant obtained from prior studies.

EXPERIMENTAL

The program included two lab-scale demonstrations of key stages of the reaction. These two demonstrations were performed at slightly larger scale and in instrumented equipment to better understand the chemistry. The lab-scale demonstrations focused on TPB decomposition and offgas generation.

No peroxide-free experiment (peroxide blank) was completed to demonstrate that the decomposition was due to the addition of hydrogen peroxide and not the elevated temperatures. A previous experiment completed at a lower temperature (45°C), lower copper concentration (24 mg/L), and lower peroxide addition rate (half the rate) in the same equipment led to minimal TPB decomposition (10).

Figure 1 shows a schematic of the equipment. The simulant was added to a 1-liter carbon steel vessel with glass lid. The slurry was stirred using an overhead stir motor and agitator blades. The temperature was controlled using a silicon oil heating bath. The hydrogen peroxide was metered in using a syringe pump. A pH probe monitored the pH during the reaction. Mass flow controllers supplied a 15.6 cc/min nitrogen and 4.9 cc/min air purge to duplicate the HLW tank's nitrogen purge with tank air inleakage. The offgas flowed through a process water cooled condenser (process water temperature varied from 17–22°C) to minimize the moisture content in the offgas and minimize the dewatering of the slurry. A sample from the offgas downstream of the condenser was pulled each hour and analyzed using an Agilent micro gas chromatograph (GC) for benzene, hydrogen, nitrogen, and oxygen.

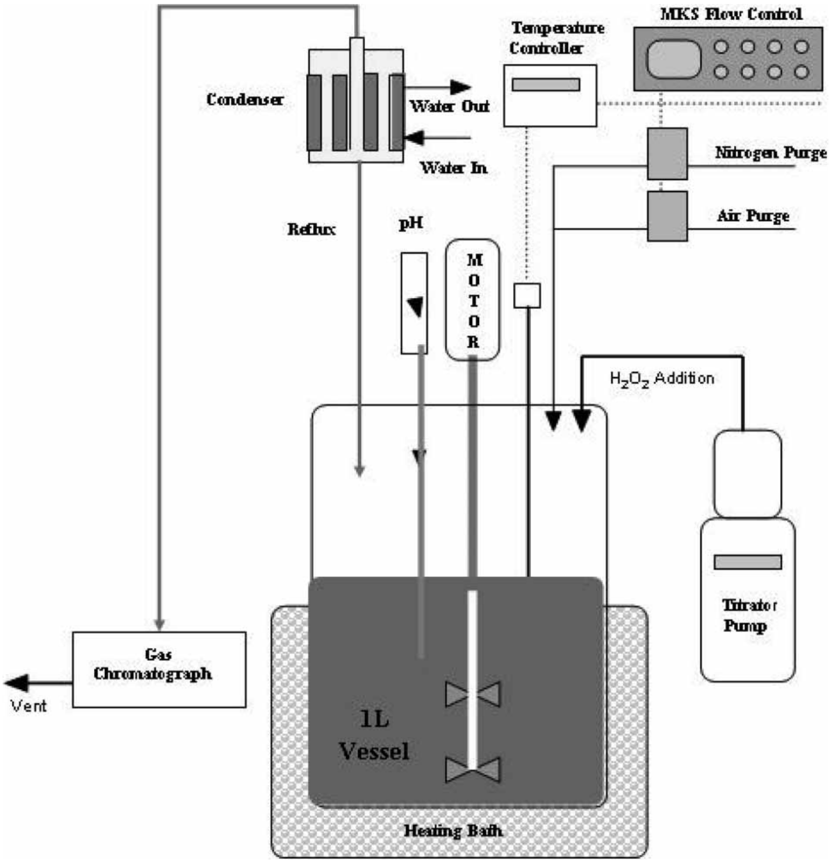

Figure 1. Lab-scale demonstration system.

Flammability in the HLW tank is currently controlled by minimizing the concentration of fuel (benzene) and oxidant (oxygen). To measure the oxygen and benzene concentration during the experiments, two gas chromatographs (Agilent microGCs) were used for offgas analysis both with two columns. The first column is a molecular sieve (Agilent G2801-60510 MolSieve 5A PLOT, 10 m 0.32 mm) to analyze for hydrogen, oxygen, and nitrogen using a thermal conductivity detector. The second column is an organic column (Agilent OV-73) to analyze for benzene, using a thermal conductivity detector. Identical methods were set up for both GCs. Each GC was calibrated using calibration standards for hydrogen, nitrogen, oxygen, and benzene before the experiments began. A calibration check occurred for each GC after 3 weeks. In addition, the GCs were baked out twice during testing to remove moisture and carbon dioxide that accumulated in the columns. The GCs were set up to measure the offgas composition from each vessel each hour. The sample pump ran for 90 seconds prior to each injection to ensure each sample was representative.

Two tests were completed in parallel lab-scale apparatus. Each test contained 600 mL of simulant contained in the 1-liter carbon steel vessel. One slurry had an initial pH of 11 and the other a pH of 14. 24 mg/L of cupric sulfate was added to the simulant before the peroxide addition began and an additional 467 mg/L of cupric sulfate was added approximately three weeks into testing. The slurry was unheated for the first week, and then the temperature was increased and held first at 35°C, then 45°C, then 55°C, and finally 75°C. Each vessel was sampled until the completion of the test. Each sample was filtered and archived. Approximately six weeks into testing, the final slurry sample was collected.

A subset of these samples was selectively analyzed for soluble K and B by ICP-ES to determine the TPB decomposition rate and estimate the benzene generation rate. The final slurry was analyzed by high-performance liquid chromatography (HPLC) for the typical TPB decomposition products (i.e., TPB, 3PB, 2PB, 1PB, and phenol).

RESULTS

The primary objective of the testing was to measure the extent of TPB decomposition. In addition, the liquid and vapor were analyzed to understand and quantify the decomposition products present during the reaction. At the completion of the testing, measurements provided a gross measure of the general corrosion rate to the carbon steel reaction vessel.

Two methods were used to determine the extent of TPB decomposition. The first analytical method utilized HPLC to directly measure the TPB ion concentration. The second method looked for the ingrowth of K and B in filtered samples to track the decomposition of the TPB.

For the pH 11 experiment, the final slurry TPB concentration was less than 10 mg/L, the detection limit for this sample. This indicates that >99.8% of the TPB was destroyed during the testing. For the pH 14 experiment, the final slurry TPB concentration was 1,310 mg/L compared to a starting concentration of 1,690 mg/L. This equates to 22% TPB decomposition for the pH 14 experiment.

For the pH 11 experiment, the K concentration in the filtrate exceeded the K concentration in the slurry for a calculated 100% TPB decomposition. The calculated TPB decomposition was 95% based on the B analyses. These analyses confirm the HPLC decomposition calculation in the pH 11 experiment. Note 48.0% of the K and 32.7% of the B was soluble in the slurry before testing began. The increase in soluble K and B was used to calculate the % TPB decomposition. Table 1 summarizes the data from the pH 11 experiment.

Table 2 shows the results for the pH 14 experiment. For the pH 14 experiment, 4–10% decomposition was noted based on the filtrate and slurry

Table 1. TPB decomposition based on results of pH 11 filtrate and slurry samples pulled at completion of testing

Sample ID analysis	Filtrate	Digested slurry	Predicted slurry composition	% TPB decomposition
ICP-ES B, mg/L	345	363	403	95
ICP-ES K, mg/L	1470	1320	1550	100
AA K, mg/L	1550	NA	1550	100

values. The percentage decomposition is calculated by comparing the filtrate concentration to the predicted slurry composition. The prediction is used as the measured slurry TPB concentration was significantly lower than expected, likely due to a slurry sample that was low in insoluble solids.

A single HPLC analysis of a sample pulled at the end of the experiment gives a snapshot of the TPB and TPB decomposition products present in that sample. For the pH 11 experiment, the concentration of 13 TPB decomposition products was each less than 10 mg/L, the detection limit for this sample. As a result, both TPB and all of the typical decomposition products were not detected. If any of these species formed, they decomposed before the sample was pulled. The one exception is biphenyl, a solid that sublimes. No biphenyl was left in the liquid but biphenyl crystals formed on the colder vessel lid and slowly migrated up the offgas line. By the completion of the run, the biphenyl crystals had "disappeared" from the rig and offgas lines. Besides TPB, the slurry also began the test with approximately 700 mg/L of phenol. Both tests destroyed phenol. All thirteen typical TPB decomposition products in a slurry sample analyzed at the conclusion of testing (triphenylboron, 2PB, phenylboronic acid, phenol, nitrobenzene, nitrosobenzene, 4-phenylphenol, 2-phenylphenol, diphenylamine, biphenyl, o-terphenyl, m-terphenyl, and p-terphenyl) were less than the 10 mg/L detection limit.

A single gas chromatography—mass spectroscopy (GC-MS) analysis of a slurry sample (all previous samples were filtered so no organic analysis was requested) pulled at completion gives a snapshot of the volatile and

Table 2. TPB decomposition based on results of pH 14 filtrate and slurry samples pulled at completion of testing

Sample ID analysis	Filtrate	Corrected starting filtrate composition	Predicted slurry composition	% TPB decomposition
ICP-ES B, mg/L	50.9	6.54	389	10.3
ICP-ES K, mg/L	376	263	1687	7.9
AA K, mg/L	321	NA	1687	4.1

semivolatile organic species present in that sample. The pH 11 slurry was analyzed by two methods to search for any remaining semivolatile or volatile organic species. No organic species were identified using volatile organic analysis (VOA), including benzene. Three organic species were identified using semi volatile organic analysis (SVOA): p-terphenyl at 11 mg/L, quaterphenyl at 3 mg/L, and 3-dimethylamino phenol at 2 mg/L.

At the completion of the test, a single sample was pulled to determine the total inorganic carbon (TIC) and total organic carbon (TOC) content. The slurry was analyzed by two methods to search for any remaining inorganic (primarily carbonate) or organic species. The pH 11 slurry had a TIC of 2760 mg/L and a TOC of 1160 mg/L (82.3 % organic carbon destruction). The measured TIC is equivalent to 0.230 M carbonate if the entire TIC is carbonate.

A single analysis of a sample pulled at completion measured the anions present in that sample. Formate and oxalate are two of the anions in the IC analytical suite. Neither formate nor oxalate is added as part of the simulant so any oxalate or formate present at the end apparently formed during testing, although it also could have formed during the prior hydrolysis experiment. Formate is 26.7% C by mass and oxalate is 27.3% C by mass. The calculated carbon mass from formate and oxalate is 1030 mg/L. This agrees reasonably with the 1160 mg/L TOC measured by TIC/TOC. It should also be noted that the large increase in nitrate (comparing pH 14 to pH 11) is due to the acidification of the sample with nitric acid. Also, about half of the nitrite, a corrosion inhibitor, was consumed (either due to acidification or oxidation) during testing. Table 3 contains the results.

A pH probe was operational throughout the pH 11 experiment. Filtrate samples were analyzed for pH as a confirmation for both experiments. The calibration of the pH probe slowly degraded during testing. No attempt was made to recalibrate as that would upset the offgas measurements. The pH dropped as low as pH 9.1 during the pH 11 experiment. Much of the pH drop likely resulted from the two additions of copper catalyst (a 467 mg/L

Table 3. Anion results

Analyte	pH 11	pH 14	Units
F^-	<13	<19	mg/L
HCO_2^-	424	162	mg/L
Cl^-	94	136	mg/L
NO_2^-	8,600	15,400	mg/L
Br^-	<100	<100	mg/L
NO_3^-	72,800	11,100	mg/L
PO_4^{3-}	391	1,210	mg/L
SO_4^{2-}	1,150	1,160	mg/L
$C_2O_4^{2-}$	2,580	1,130	mg/L

$CuSO_4 \cdot 5H_2O$ concentration has a pH of 4.0). The pH profile resembled that expected for a Fenton reaction, where the pH drops due to the production of organic acids and later rises due to destruction of these organic acids (5). While pH can be controlled through the addition of sodium hydroxide during processing, no attempt was made to control pH in these experiments. The pH remained above 14 throughout the pH14 experiment. Figure 2 displays the pH data for the pH 11 experiment.

Analysis for benzene is needed to quantify the benzene produced and assess flammability issues. The lower flammability limit (LFL) for benzene is 1.2 vol % in air (9). At a benzene concentration less than 1.2 vol %, insufficient fuel exists to support combustion. The benzene concentration was below 25% of the LFL throughout the testing. At pH 11, benzene spikes occurred while increasing the temperature from 35°C to 45°C, from 45°C to 55°C, and from 55°C to 75°C. The maximum benzene concentration occurred immediately after the temperature increased from 55°C to 75°C due to the increased mass transfer of benzene from the slurry to vapor. However, the steady-state benzene concentration quickly dropped back to a concentration of well less than approximately 100 ppm_v, which corresponds to 2 mg/L/hr benzene. These peaks are influenced by the rate of heating and would likely have been much shorter and broader for a slow temperature ramp, similar to what would be expected in The HLW tank. Figure 3 contains the data for the pH 11 experiment.

A running mass balance was maintained throughout the experiments since the reactor volume changed from peroxide additions and sample removal. The

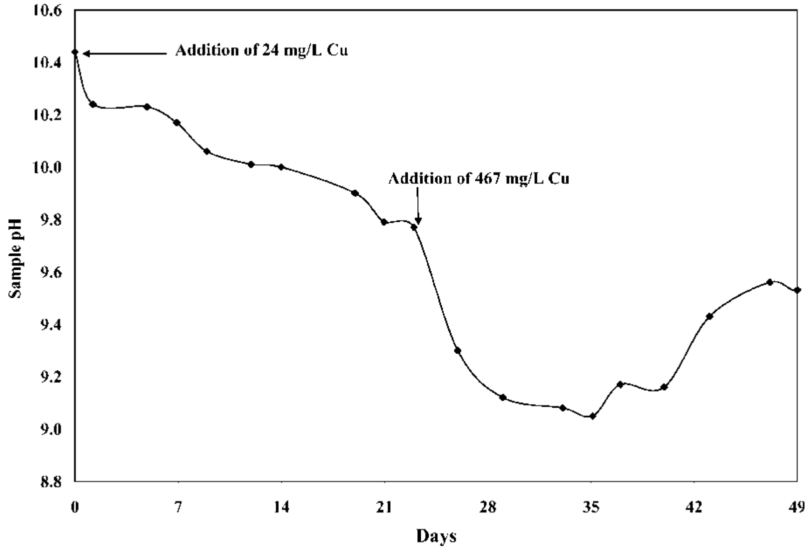

Figure 2. pH profile throughout pH 11 experiment.

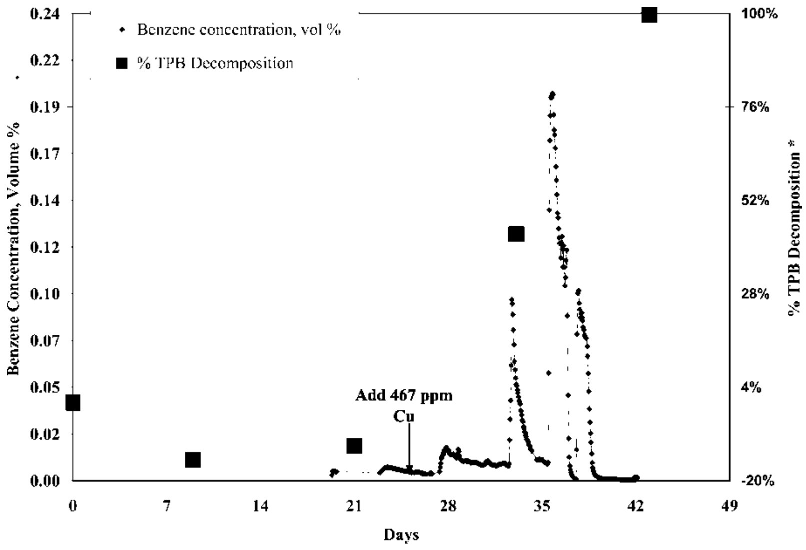

Figure 3. Benzene concentration and TPB decomposition (calculated from K^+ analysis) during pH 11 experiment.

combined impact is a slow dilution of the slurry. The concentrations of TPB, B, and K throughout the experiment are corrected for the dilution. One of the most compelling advantages of an oxidation process is minimization of benzene generation during decomposition of the tetraphenylborate. Slurry samples analyzed at the completion of testing and offgas data were used to identify and quantify the TPB decomposition products. Ideally, all of the TPB would decompose to carbon dioxide. However, carbon dioxide was not analyzed in the offgas. Table 4 summarizes the information for the pH 11 experiment.

The data demonstrate that no detectable TPB, benzene, biphenyl, or phenol was present in the slurry post reaction. Small quantities of p-terphenyl, quaterphenyl, and phenol, 3-dimethylamino were present just above their detection limits and accounted for <0.3% of the decomposition products. Two major species produced were oxalate and formate. It also demonstrates that 5.4% of the organic carbon decomposed to benzene gas. The remainder of the organic species likely decomposed to CO and CO_2 (did not analyze for CO or CO_2 in offgas).

At pH 14, benzene spikes occurred while increasing the temperature from 35°C to 45°C, and from 45°C to 55°C. The maximum benzene concentration occurred immediately after the temperature increased from 55°C to 75°C due to the increased mass transfer of benzene from the slurry to vapor and possibly increased reactivity of the intermediates. However, the steady state benzene concentration soon dropped back to a concentration of well less than 2 mg/L/hr benzene (~100 ppm_v benzene). Figure 4 shows the data from the pH 14 test.

Table 4. pH 11 carbon balance

Component	Before (C mass, g)	After (C mass, g)	Change (C mass, g)	Amount present in final slurry as % original carbon
TPB	6.32	0.00	−6.32	0
Biphenyl	0.36	0.00	−0.36	0
Phenol	0.45	0.00	−0.45	0
Total organic carbon	7.13	0.00	−7.13	0
Formate	0.00	0.20	0.20	2.8
Oxalate	0.00	1.26	1.26	17.7
Benzene (g)	0.03	0.42	0.39	5.4
Carbonate	3.31	3.90	0.60	8.4
p-Terphenyl	0.00	0.01	0.01	0.2
Quaterphenyl	0.00	0.00	0.00	0.1
Phenol, 3-dimethylamino	0.00	0.00	0.00	0.0
Total	10.47	5.80	−4.66	34.6

Analysis for oxygen is needed to quantify the oxygen produced due to decomposition of hydrogen peroxide and assess flammability issues (i.e., whether minimum oxidant control is sustained during the reaction). The air and nitrogen purge provided 5% oxygen and 95% nitrogen to the inlet of the

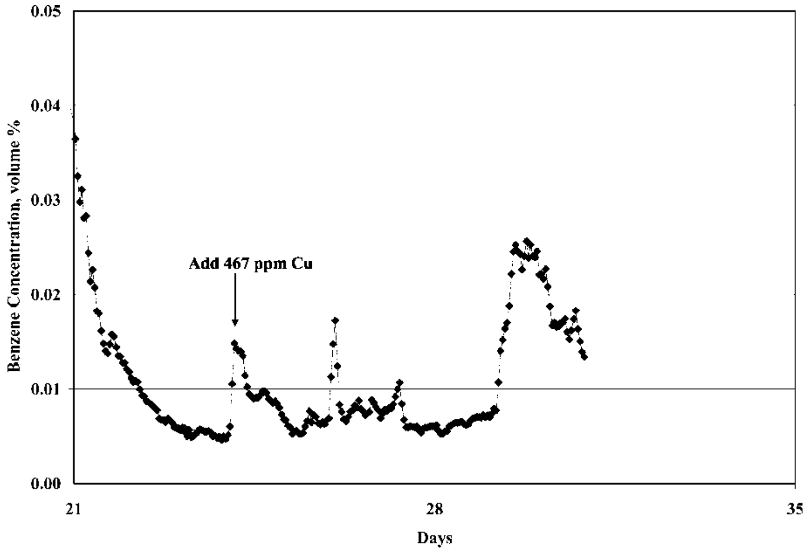

Figure 4. Benzene concentration during portion of pH 14 experiment.

vessels (based on the expected air inleakage into the HLW tank). The oxygen concentration could increase to as much as 9.87 vol % if all of the hydrogen peroxide decomposed to water and oxygen. The minimum oxygen concentration (MOC) for benzene is 11.4 vol % (9). At an oxygen concentration less than 11.4 vol %, insufficient oxygen exists to support combustion. The oxygen concentration remained below 60% of the MOC throughout the testing, with the exception of a few data points taken after maintenance of the equipment introduced room air (21% oxygen) into the offgas. The oxygen concentration exceeded 6.9 vol %, the HLW tank's oxygen limit, for much of the testing. The oxygen concentration can be controlled by decreasing the hydrogen peroxide addition rate. Figure 5 shows the data for both experiments.

The addition of 1.1064 g of $CuSO_4 \cdot 5H_2O$ (0.315 g Cu added to 674 mL of slurry) led to a 467 mg/L increase in the copper concentration on day 22. The copper addition correlates strongly with the destruction of TPB for the following reasons. First, no detectable TPB decomposition occurred before the addition of copper. The soluble K and B concentration increased after addition of copper, indicating that the TPB was being consumed. Second, the offgas oxygen concentration dropped from a steady value of 7 vol % to 6 vol % after the addition of the 467 mg/L copper. If no peroxide is added, the expected oxygen concentration is 5 vol %. (i.e., the concentration entering the vessel). If none of the peroxide is oxidizing the TPB, it will react with itself decomposing to oxygen and water (explaining the 7 vol % oxygen during the early part of the testing). Upon copper addition, the oxygen concentration dropped to ~6 vol %, indicative that approximately

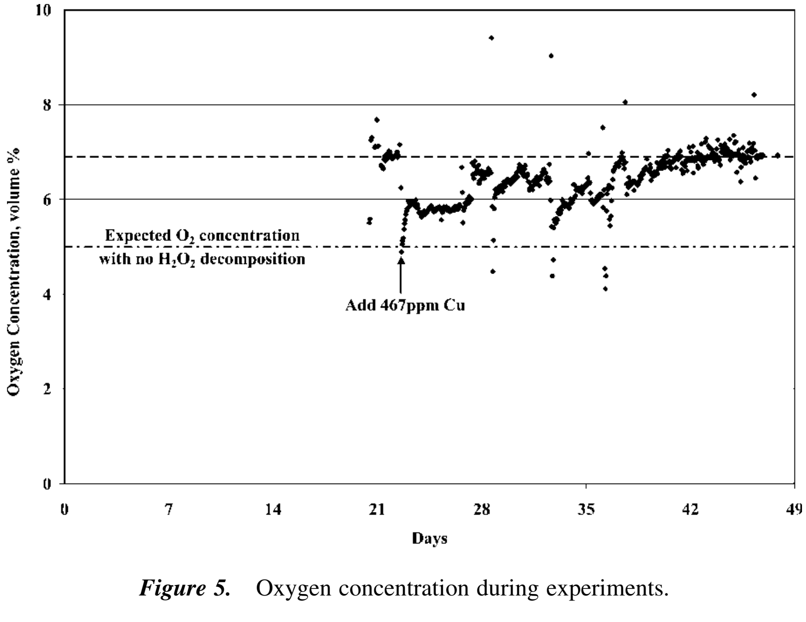

Figure 5. Oxygen concentration during experiments.

half of the peroxide was being consumed in oxidation reactions. Figure 4 summarizes the measured benzene concentration and the calculated TPB decomposition based on filtrate K analyses. Figure 5 summarizes the oxygen concentration after the addition of 467 mg/L copper. Note the drop in oxygen concentration after the additional copper addition, indicative of more efficient peroxide utilization.

The corrosion of the carbon steel vessels was measured at the end of each experiment. The general corrosion rate is 3.7 mils/year for the pH 11 experiment and 1.6 mils/year for the pH 14 experiment. The demonstrations used carbon steel reaction vessels to serve as crude corrosion coupons. Each vessel was fabricated by welding a 3-1/2″ schedule 10 carbon steel pipe cap to 31/2″ schedule 10 carbon steel pipe. Three internal baffles improved mixing. The inside carbon steel surfaces were not polished before testing as is traditional with corrosion coupons. The outside of each vessel was painted with Krylon paint to prevent corrosion during exposure in the silicon oil bath. The corrosion measurement followed ASTM G1-901 (13).

Previous corrosion testing relied on electrochemical tests of carbon steel (Type III waste tank steel is ASTM A537 class 1) test specimens in simulated waste solutions (14). Tests at pH 11 and between 45 and 65°C revealed a relatively low general corrosion rate of 5 mils per year (0.005 inches per year). The threshold for reporting wall-thinning in the in-service inspection program for high-level waste tanks is 10% of wall thickness (e.g., 50 mils' loss in 0.5-inch-thick plate). The 3.7-mil/y loss would also not significantly affect the cooling coils, whose wall thickness is about 150 mils (14). The experiments did not provide a measure of pitting corrosion because the original carbon steel vessels were not sanded or polished and contained numerous pits.

CONCLUSIONS AND RECOMMENDATIONS

A copper catalyzed, peroxide oxidation of TPB process was demonstrated in two lab-scale experiments with simulant starting at pH 11 and pH 14. The experiment at pH 11 led to complete TPB decomposition (i.e., $>99.8\%$ TPB decomposition complete in <3 weeks). The experiment resulted in an approximately 75% increase in waste volume. The experiment at pH 14 led to slower TPB decomposition—approximately 15% decomposition in 9 weeks. The volume roughly doubled during testing, so this process is not feasible in a HLW tank without a better catalyst system.

The oxidation process may be useful for applications that require the pH to remain >7, yet need to oxidize organic species. Several issues remain which must be resolved prior to implementation of this technology.

1. The test at pH 11 used a waste simulant. Future testing needs to be completed with actual waste to confirm these findings.

2. The test at pH 11 used a single peroxide addition rate (chosen to minimize testing time), at two copper catalyst concentrations, and at a pH lower than targeted.

Further development of a copper catalyzed tetraphenylborate oxidation process is recommended. The issues identified above should be resolved through the following steps.

1. Develop an optimized copper catalyzed tetraphenylborate oxidation flowsheet. Measure CO, CO_2, benzene, and oxygen in the offgas.
2. Conduct a confirmatory test for the pH 11 experiment with actual waste.
3. Determine corrosion rate and measure pitting potential in carefully controlled corrosion experiments.

The experiments should focus on pitting potential of the carbon steel tank and cooling coils.

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