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**Advanced Oxidation
Oxalate Decomposition Testing with Ozone - 12534**

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ABSTRACT

At the Savannah River Site (SRS), oxalic acid is currently considered the preferred agent for chemically cleaning the large underground Liquid Radioactive Waste Tanks. It is applied only in the final stages of emptying a tank when generally less than 5,000 kg of waste solids remain, and slurring based removal methods are no-longer effective. The use of oxalic acid is preferred because of its combined dissolution and chelating properties, as well as the fact that corrosion to the carbon steel tank walls can be controlled.

Although oxalic acid is the preferred agent, there are significant potential downstream impacts. Impacts include:

- Degraded evaporator operation.
- Resultant oxalate precipitates taking away critically needed operating volume.
- Eventual creation of significant volumes of additional feed to salt processing.

As an alternative to dealing with the downstream impacts, oxalate decomposition using variations of ozone based Advanced Oxidation Process (AOP) were investigated. In general AOPs use ozone or peroxide and a catalyst to create hydroxyl radicals. Hydroxyl radicals have among the highest oxidation potentials, and are commonly used to decompose organics. Although oxalate is considered among the most difficult organic to decompose, the ability of hydroxyl radicals to decompose oxalate is considered to be well demonstrated. In addition, as AOPs are considered to be “green” their use enables any net chemical additions to the waste to be minimized.

In order to test the ability to decompose the oxalate and determine the decomposition rates, a test rig was designed, where 10 vol% ozone would be educted into a spent oxalic acid decomposition loop, with the loop maintained at 70°C and recirculated at 40L/min. Each of the spent oxalic acid streams would be created from three oxalic acid strikes of an F-area simulant (i.e., Purex = high Fe/Al concentration) and H-area simulant (i.e., H area modified Purex = high Al/Fe concentration) after nearing dissolution equilibrium, and then decomposed to ≤ 100 Parts per Million (ppm) oxalate. Since AOP technology largely originated on using ultraviolet (UV) light as a primary catalyst, decomposition of the spent oxalic acid, well exposed to a medium pressure mercury vapor light was considered the benchmark. However, with multi-valent metals already contained in the feed, and maintenance of the UV light a concern; testing was conducted to evaluate the impact from removing the UV light. Using current AOP terminology, the test without the UV light would likely be considered an ozone based, dark, ferrioxalate type, decomposition process.

Specifically, as part of the testing, the impacts from the following were investigated:

- 1) Importance of the UV light on the decomposition rates when decomposing 1 wt% spent oxalic acid
- 2) Impact of increasing the oxalic acid strength from 1 to 2.5 wt% on the decomposition rates
- 3) For F-area testing, the advantage of increasing the spent oxalic acid flowrate from 40 L/min (liters/minute) to 50 L/min during decomposition of the 2.5 wt% spent oxalic acid

The results showed that removal of the UV light (from 1 wt% testing) slowed the decomposition rates in both the F & H testing. Specifically, for F-Area Strike 1, the time increased from about 6 hours to 8 hours. In H-Area, the impact was not as significant, with the time required for Strike 1 to be decomposed to less than 100 ppm increasing slightly, from 5.4 to 6.4 hours. For the spent 2.5 wt% oxalic acid decomposition tests (all) without the UV light, the F-area decompositions required approx. 10 to 13 hours, while the corresponding required H-Area decompositions times ranged from 10 to 21 hours. For the 2.5 wt% F-Area sludge, the increased availability of iron likely caused the increased decomposition rates compared to the 1 wt% oxalic acid based tests. In addition, for the F-testing, increasing the recirculation flow rates from 40 liter/minute to 50 liter/minute resulted in an increased decomposition rate, suggesting a better use of ozone.

INTRODUCTION

At the Savannah River Site closure the single shell radioactive liquid waste tanks are being readied for closure. They were constructed from commercial grade carbon steel in the mid-1950's to the early 1960's. In general, their dimensions range from 23 to 26 meters in diameter, 7 to 10 meters high, and the volumetric capacity ranges from 2.8 to 4.9 million liters. Typically, each tank contains an internal labyrinth of cooling coils made from carbon steel, which further complicates waste removal and cleaning (1).

Savannah River Site has utilized both mechanical and chemical methods for removing the waste from tanks. The mechanical means, predominantly consisting of mixing/slurrying with ensuing transfer, are used to remove the bulk of the waste. Chemical cleaning is deployed only when residual quantities of solids remain (e.g. less than about 5,000 kg of solids per tank) and mechanical methods are no-longer effective. Chemical cleaning is necessary to support the stringent requirements for closure. The closure requirement for the tanks is primarily driven by the high specific activity of the waste, large interior contaminated surface areas (including the coils), and close proximity to the water table (1).

Oxalic acid remains the preferred acid for cleaning the metal surfaces because of its combined digesting and chelating effects. Other common acids considered for cleaning include nitric acid and oxalic/citric acid blends. Oxalic acid is preferred over nitric acid due to corrosion concerns; oxalic acid forms a passivation layer on carbon steel surfaces. While both pure oxalic acid and oxalic/citric acid mixtures have been shown to be equally effective in dissolving waste residuals, there are undesirable downstream impacts associated with introducing citric acid into the radioactive liquid waste system. For chemical cleaning, the current process uses 8 wt% concentrated oxalic acid as the solvent. During the current chemical cleaning process, in three steps, a total of about 380,000 liters of 8 wt% oxalic acid solution is added to the Treatment Tank and slurried. After allowing adequate time for the acid to react with the residual solids, mixing is stopped, the transferable solids/spent acid is transferred out of the Treatment Tank into an existing liquid radioactive waste tank pre-staged with adequate quantities of concentrated supernatant to restore the free hydroxide of the waste stream to within radioactive liquid waste system corrosion control parameters.

Because of the sodium concentration of concentrated supernatant (e.g. Na >> 5M) and the amount of acid added, large quantities of sodium oxalate would precipitate out as a solid. Modeling shows that for every tank treated, about 51,000 kg of new sodium oxalate solids will be formed for feed to vitrification at DWPF (Defense Waste Processing Facility). Regardless of the handling strategy, most would wash out creating 1.9 million liters of Salt Processing feed will be created (10).

A promising alternative to bulk oxalic acid cleaning will utilize a more dilute oxalic acid (OA) (nominally about 1 or 2.5 wt%, at a pH of around 2.0). It is planned to be coupled to an advanced oxidation technology to decompose the resultant metal oxalate/spent cleaning solutions. As the metal oxalate is decomposed through hydroxylation, metal oxide precipitates form, while the oxalate is decomposed and offgassed as carbon dioxide. A simplified process flowsheet which minimizes these impacts is shown as Figure 1.

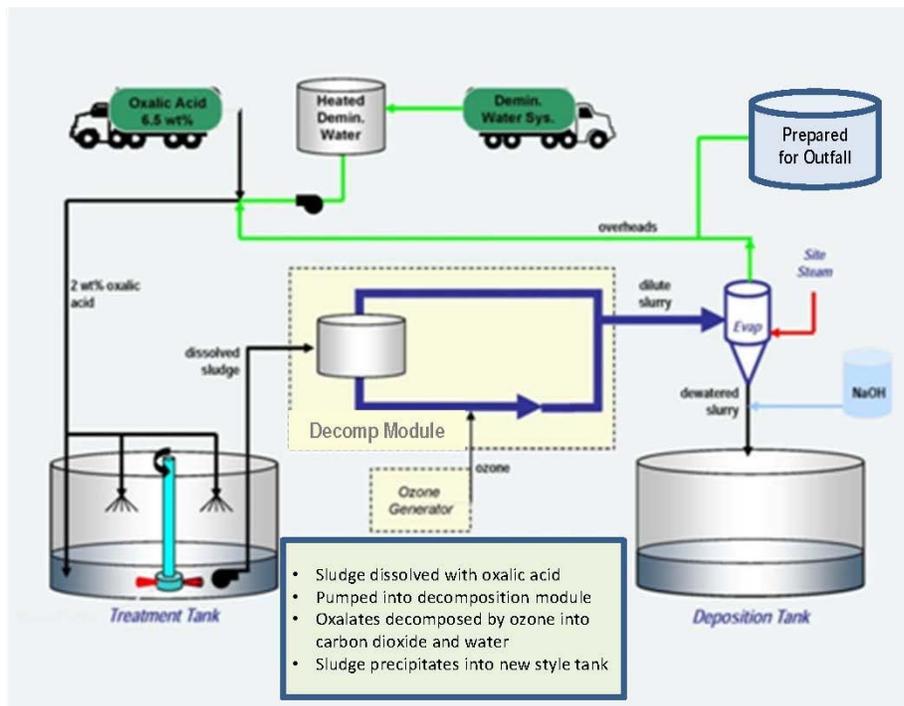


Figure 1. Simplified Process Flow Diagram of the Decomposition Process (1)

To achieve maximum dissolution effectiveness, the pH of the Treatment Tank would be maintained near 2.0 throughout the cleaning by oxalic additions. To maximize solid suspension so that material can be transferred out of the Treatment Tank additional mixers would be added. The spent oxalic acid solution/metal oxalate slurry from the Treatment Tank would be transferred to the oxalate decomposition reactor. The oxalate decomposition reactor is recirculation loop where ozone is forced into the slurry to decompose the oxalates. To minimize differences in the decomposition rates, originally UV light was installed to catalytically aid in formation of hydroxyl radicals. The primary reaction for the creation of hydroxyl radicals from ozone and UV is shown by Reaction 1 (2, 3).



From a UV light perspective, the solids can limit the ability of the UV to create hydroxyl radicals, either by limiting the ability of the light to penetrate the solution, or by increasing the fouling rate on the UV light. Both ultimately can decrease the overall catalytic effectiveness of the UV, thereby decreasing the concentration of formed hydroxyl radicals. An example of UV light test fouling is shown by Figure 2 (2).

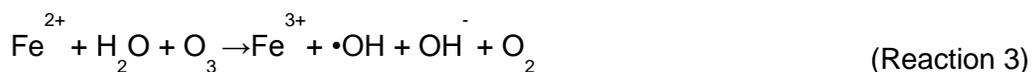
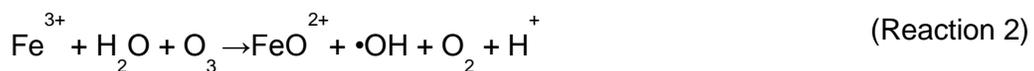


Figure 2. Fouling of the UV Light Apparatus



Figure 3. Oxalate Solutions Throughout Decomposition

Although the effectiveness of the UV would be decreased due to the increased metals (either through plating or through the ability of the light to penetrate the solution), hydroxyl radicals are also created from Fenton-type “dark catalytic” reactions. That is, hydroxyl radicals would continue to be produced because of the presence of ozone and other catalysts. Other reactions in the decomposition loop which can produce hydroxyl radicals include homogenous catalytic type reactions. The Fenton’s type reactions will compete for the intermediate peroxide. The possible Fenton type reactions are shown by Reactions 2 and 3.



EXPERIMENTAL

Simulant Recipe

Largely based on verifying known sludge chemical dissolution characteristics, and known tank characterizations, simulant recipes were developed for the F-Area and H-Area sludge dissolution testing. The physical properties are shown in Table 1, while the compositions are shown in Table 2 (9).

Table I. Physical Properties of the Simulants (9)

Property	F-Area Simulant	H-Area Simulant
Density, g/ml	1.119	1.148
wt% total solids	13.55	17.2
wt% soluble solids	3.05	2.00
wt% insoluble solids	10.5	15.2
pH	12.32	12.54

Table II. Compositions of the Simulants (9)

Species	F-Area Simulant		H-Area Simulant	
	Concentration mg/l	wt%	Concentration mg/l	wt%
Ag	52	0.03	<172	<0.10
Al	10105	6.64	71935	36.42
Ba	338	0.22	506	0.26
Ca	3310	2.18	1318	0.77
Cd	<22	<0.010	<23	<0.10
Ce	340	0.22	135	0.07
Cr	350	0.23	140	0.07
Cu	173	0.11	102	0.05
Fe	35383	23.25	6176	3.13
K	189	0.12	290	0.15
La	205	0.13	67	0.03
Li	179	0.12	153	0.08
Mg	155	0.10	1193	0.60
Mn	4568	3.00	6435	3.26
Mo	None Added	None Added	<23	<0.10
Na	13718	9.01	8532	4.32
Nd	None Added	None Added	47	0.02
Ni	4025	2.65	626	0.32
P	63	0.04	25	0.01
Pb	142	0.09	<172	<0.10
S	469	0.31	79	0.04
Si	1307	0.86	302	0.15
Sr	118	0.08	35	0.02
Ti	None Added	None Added	<23	<0.01
Zn	412	0.27	79	0.04
Zr	612	0.40	253	0.13
Hg	1757	1.16	3221	1.63
F ⁻	<112	<0.08	<100	<0.06

Cl ⁻	1673	1.10	<100	<0.06
NO ₂ ⁻	10183	6.69	695	0.40
NO ₃ ⁻	2965	1.95	3730	2.17
H ₂ O	NA	39.04	NA	45.86

Note: H₂O refers wt% of interstitial liquid in formed product

Process Equipment

While designing the test setup, the equipment associated with the decomposition testing was grouped as either being associated with either:

- 1) Dissolution (i.e., making and supplying the spent oxalic acid/metal oxalate slurry feed to the decomposition unit), or
- 2) Decomposition (i.e., making/transporting the ozone to the recirculation loop, and decomposing the oxalate).

Dissolution System

1. *Dissolution Tank* - The dissolution tank was a 30 gallon (approx. 114 liters) stainless steel drum. The tank was loaded with approximately 20 cm of sludge simulant and then treated with subsequent charges of oxalic acid in order to generate feed for the decomposition process.
2. *Drum Band Heaters* - Drum band heaters were positioned on the dissolution tank to control the temperature to 70°C ± 5°C.
3. *Hand Held Transfer Pump* – A hand held transfer pump was used to transfer the solution from the dissolution vessel to the decomposition loop.
4. *Instrumentation* - Temperature and pH probes were installed in the system for monitoring the system throughout the dissolutions.

Decomposition System

- 1) *Decomp Loop* – The loop was the main reaction chamber for the decomposition process. It had a volume of slightly more than 60 liters. The ozone injection point was immediately upstream from a 10 liter decomp, which was installed to provide some additional residence time for the decomposition. The decomposition vessel was also operated at 70°C ± 5°C and 10 ±2 psig.
- 2) *Inline Pump* – The inline pump provided driving head to move the solution around the loop.
- 3) *Back Pressure Valve* - The back pressure valve was throttled so that the pumping action created an approximate 10 psig overpressure on the system. This overpressure enabled more ozone to be maintained in solution.
- 4) *Oxygen Concentrator* - Separated oxygen from compressed air to provide oxygen feed to the ozone generator.
- 1) *Ozone Generator*- Received enriched 90-95% oxygen from the concentrator and used a corona discharge to convert a portion to ozone. The ozone was approximately 8-12% by weight of the gas exiting the generator. The output pressure of the ozone generator was generally 14-20 psig, capable of providing an ozone injection rate of 60 gram/hour based on replicating previous testing. The ozone provided the oxidant for making the radicals necessary for the decomposition of the oxalates into carbon dioxide.

- 2) *Heater* - The heater provided a heat source to maintain the solution at the process temperature of $70^{\circ}\text{C} \pm 5^{\circ}\text{C}$.
- 3) *UV or UV Light* - Based on previous testing, a UV ~270 nm wavelength light was installed. In theory, when energized it would aid in the creation of hydroxyl radicals. The overall design of the light was based on maximizing the effectiveness of the UV.
- 4) *Instrumentation* - Several different instruments were utilized to provide online indication of key process parameters. These included solution flow rate, oxygen feed rate to ozone generator, ozone feed concentration, pressure, temperature, gas flow rate, pH, and oxidation reduction potential (ORP).

Ozonation Design

The solubility of ozone in analogous previous testing was determined to be 38.8 parts per million (ppm) (5), while the theoretical solubility using Henry's Law can be approximated as 31.2 ppm. To provide additional conservatism, the test design assumed an ozone solubility of about 25 ppm. The decomposition loop recirculation rate needed to achieve 25 ppm ozone, at the injection point using an ozone injection rate of 60 gram/hour, was therefore calculated to be 40 L/min.

The "efficiency" of the decomposition process, as measured by the ratio of moles of ozone injected to moles of oxalic acid decomposed, was also considered in the design basis. Based on the results of the previous testing, a conservative process efficiency of 33%, or 3 moles of ozone injected per mole of oxalic acid decomposed, was assumed for the process design (5).

Testing Scope

Spent 1 wt% Oxalic Acid Decomposition Testing

For the 1 wt% oxalate decomposition testing, testing was performed with the UV operated under two separate conditions. They were decomposition with no light and decomposition with the UV light maintained (i.e., clean).

Based on previous testing, each sequence required three dissolution strikes followed by decomposition. That is, a minimum of three applications of fresh 1 wt% oxalic acid solution or strikes were required to adequately dissolve the simulants and achieve the target application pH during the dissolution step of less than 2.0. Based on the different conditions tested and the required minimum of three strikes, a total of 12 strikes were completed for the 1 wt% oxalate decomposition testing. Table 3 shows the test matrix with the appropriate strike designations.

Table III. Spent 1 wt% Oxalic Acid Decomposition Testing Matrix

Simulant	UV Light Condition	Strike 1	Strike 2	Strike 3
F-Area	No Light	F-NL-1	F-NL-2	F-NL-3
	Clean Light	F-CL-1	F-CL-2	F-CL-3
H-Area	No Light	H-NL-1	H-NL-2	H-NL-3
	Clean Light	H-CL-1	H-CL-2	H-CL-3

Spent 2.5 wt% Oxalic Acid Decomposition Testing

This was similar to the spent 1 wt% Oxalic Acid Decomposition Testing, but only included the no UV light and was performed using 2.5 wt% oxalic acid. It was conducted to directly evaluate the impact of an increase in oxalic acid concentration on the effectiveness of the decomposition process. This was performed because a 2.5 wt% oxalic acid solution has the advantage of removing a greater quantity of sludge per solvent application, thus reducing the required number of applications. This task was used to demonstrate that the decomposition process could be completed efficiently with a higher solvent concentration and no UV light, without significantly increasing duration. A total of six strikes were completed for this phase of testing. Table 4 shows the test matrix with the appropriate strike designations.

Table IV. Spent 2.5 wt% Decomposition Testing Matrix

Simulant	Test	Strike 1	Strike 2	Strike 3
F-Area	No UV Light	A-F-NL-1	A-F-NL-2	A-F-NL-3
H-Area	No UV Light	A-H-NL-1	A-H-NL-2	A-H-NL-3

Increased Recirculation with Spent 2.5 wt% Oxalic Acid Decomposition Testing

This testing increased the decomposition flowrate to 50 L/min, compared to the baseline flowrate of 40 L/min, without changing the ozone feed rate of 60 grams per. Table 5 shows the test designation matrix.

Table V. Increased Recirculation with Spent 2.5 wt% Decomposition

Simulant	Test Series	Strike 1	Strike 2	Strike 3
F-Area	No UV Light	A-F-NL-1	A-F-NL-2	A-F-NL-3

Analytical Testing

Prior to each acid strike, the mass, and moisture content of the wet sludge added to the dissolution drum were determined in order to calculate the mass of dry solids. Dissolution samples were collected periodically throughout the dissolutions and analyzed for oxalate concentration and dissolved metal concentrations. The pH of the solution was also data logged throughout each dissolution strike. The duration of each of the dissolution steps (including preparation and analysis) was approximately 24 hours.

Samples were collected periodically throughout the decompositions and analyzed for oxalate concentration (oxalic acid titration) and dissolved metal concentrations. The decomposition end point was 90% destruction of the oxalic acid, however each strike was

decomposed until ~99% destruction of oxalic acid (<100 ppm) was achieved. The oxalate concentration in the decomposition loop was analyzed by titration analysis of grab samples throughout each strike. As a way to trend the oxalate concentration over time, in-line TOC (Total Organic Carbon) analyzers were also used. The decomposition end point was 90% destruction of the oxalic acid, however each strike was decomposed until ~99% destruction of oxalic acid (<100 ppm) was achieved. Titration of the oxalic acid solutions with potassium permanganate was used to confirm the end points of each test series. The TOC analyzers were used to provide an indication of when the end point was be approached, however the reduced accuracy of the instruments at low concentrations meant that the oxalic acid titration method was still required to confirm the endpoint. The pH and TOC were also included in the data collected throughout each decomposition strike using in-line analyzers.

RESULTS

Decomposition Rates

Table 6 shows the initial oxalate concentration and the time required for the oxalate to be decomposed to less than 100 ppm.

Table VI. Decomposition Rates

Task	Strike #	OA (ppm)	Required Decomposition Duration (hr)
2.5 wt%	A-F-NL-1	21558	13.2
	A-F-NL-2	22060	12.3
	A-F-NL-3	22510	12.3
	A-H-NL-1	22735	20.3
	A-H-NL-2	24311	17.3
	A-H-NL-3	25211	21.3
Increased Recirculation	B-F-NL-1	22960	10.6
	B-F-NL-2	23860	10.1
	B-F-NL-3	24311	11.3
1 wt% F-Area	F-NL-1	8156	8.0
	F-NL-2	10093	8.5
	F-NL-3	9553	7.6
	F-CL-1	6804	5.0
	F-CL-2	7886	5.6
	F-CL-3	8877	6.2
1 wt% H-Area	H-NL-1	59930	6.1
	H-NL-2	9598	6.1
	H-NL-3	10048	6.0
	H-CL-1	66240	5.1
	H-CL-2	7255	5.6
	H-CL-3	7570	5.0

The results showed that the average duration of the spent 2.5 wt% oxalic acid decomposition testing was only 1.4 times longer than the 1 wt% no UV. Therefore, increasing the oxalic acid concentration to 2.5 wt% has a positive impact on the decomposition rate.

The increased recirculation test at 2.5 wt% also decreased the duration. The H-Area results for 2.5 wt% did not show the same drastic improvement as the F-Area results. The duration of the 2.5 wt% decomposition strikes varied over a large range from 10.4 hours to 21.3 hours. The A-H-NL-3, 2-H-2, and 2-H-three strikes had particularly long durations of 21.3, 17.1, and 20.3 hours, respectively. One explanation for the extended duration of these H-Area strikes during the 2.5 wt% OA decomposition tests is the composition of the H-Area sludge simulant, which has high aluminum and low iron concentrations compared to the F-Area simulant. Iron is known to be a catalyst for the decomposition reaction by promoting the formation of hydroxyl radicals. Although aluminum also can serve as a catalyst, it is much less effective. Based on Table 2, there are also other unique factors associated with the H-Area sludge, such as no copper added to the simulant.

Overall the decomposition testing with 2.5 wt% oxalic acid and the no UV light demonstrated the increased efficiency compared to the 1 wt% oxalic acid and the no UV light tests

CONCLUSION

The decomposition testing demonstrated the following:

- 1) Based on 1 wt%, UV light has an impact, but its removal was not detrimental to the decomposition rate
- 2) The time needed to reach the decomposition endpoint decreased as the spent oxalic acid strength to be decomposed increased from 1 to 2.5 wt%
- 3) Increasing the flowrate in the decomposition loop from 40L/min to 50L/min decreased the required time by about 20%

The decomposition rates and efficiency (i.e., moles of ozone/moles of oxalate) were determined based on testing with spent 1 wt% and spent 2.5 wt% oxalic acid, with the decomposition end point set at less than 1,000 ppm oxalate.

To conservatively confirm no UV was needed, the decomposition of spent 1 wt% acid testing was performed using maintained UV lights (i.e., cleaned), non-energized UV lights (i.e., no-UV), and degraded UV (i.e., non-maintained lights, tests only after film formed on energized UV lights from previous testing). The results showed that the decomposition rate without the UV would be acceptable. For the conditions and simulant tested at 1 wt% only about 34% more time would generally be required to decompose the spent 1 wt% acid without UV (i.e., 6 hours vs 8 hours).

For the spent 2.5 wt% oxalic acid decomposition without the UV for most decompositions approx. 10-16 hours were required, however some of the H-Area strikes required in excess of 21 hours. The increased availability of oxalate for destruction in the 2.5 wt% oxalic acid tests resulted in high initial decomposition rates compared to the 1 wt% tests.

In addition, the testing showed that increasing the recirculation flow rates from 40 liter/minute to 50 liters/minute resulted in increased decomposition efficiencies and shorter durations.

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