QUARTERLY PROGRESS REPORTS

February 1, 2012 – April 30, 2012

PROJECT TITLE: Biodegradability Enhancement of Bioreactor Landfill Leachate with Fenton Processes

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COMPLETION DATE: July 31, 2012
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INTRODUCTION

The Hinkley Center has provided critical support for exploring the concept of Bioreactor Landfills (BL) in the past two decades and nurtured the successful story of BL at the New River Regional Landfill (NRRL), in Union County, Florida. Leachate recirculation lead to accelerated waste degradation and landfill settlement; however, prolonged leachate recirculation causes the waste mass to reach its field capacity and at this stage landfill may not need any more leachate recirculation. Leachate characteristics also stabilize and the biodegradability of the leachate decreases significantly as BOD/COD ratio of leachate decreases to less than 0.10. The reduction in biodegradability reduces the effectiveness of leachate treatment using conventional biological processes and consequently increases the treatment cost. For example, in Miami-Dade County, leachate is treated at the wastewater treatment facility and causes the reclaiming wastewater treatment cost as high as $9 per gallon in 1 GDM pilot-scale water reuse plant. For the proposed 40 MGD full-scale water reuse plant, the unit cost will still be as high as $2 per gallon due to mixing of leachate with its domestic wastewater. To reduce the cost, Fenton treatment of leachate before it is mixed with the domestic wastewater may offer a great solution to this challenging problem. The current project will study the Fenton oxidation of leachate as pre-treatment to increase biodegradability of leachate for BL. Results of the study will help obtain optimal cost-effective conditions to be used for leachate treatment using Fenton oxidation.

WORK COMPLETED THIS QUARTER

Following tasks were conducted during February 1, 2012 – April 30, 2012.

- The project website (http://www.tang.fiu.edu/hinkley-center-projects/) was created.
- Literature review suggests that previous researchers have evaluated the applicability of Fenton oxidation by determining the optimum doses for specific leachate; however, in the current research the experiments are planned to obtain a generalized relationship between

Fenton oxidation efficiency at optimum doses. A detailed plan of experiments was established. Experiments were planned in two stages.

- Leachate samples were collected from a class-I landfill located in Palm Beach County.
- The leachate samples were characterized for leachate quality parameters.
- Stage-1 Experiments were conducted.
- Fenton oxidation experiments were conducted and leachate treatability for organics removal was evaluated by analyzing total organic carbon.
- Effectiveness of Fenton oxidation for biodegradability improvement was evaluated by analyzing BOD.
- A manuscript "Statistical Analysis of Optimum Fenton Oxidation Conditions for Landfill Leachate Treatment" was prepared and submitted to the peer reviewed journal Waste Management for peer review.

EXPERIMENTAL DETAILS

Experimental Plan:

The literature review suggest that the maximum organic matter removal by Fenton oxidation is obtained at optimum conditions of pH, H_2O_2/Fe^{2+}, reaction time, temperature, and reagent doses (H_2O_2/COD_0 and Fe^{2+}/COD_0) and conventionally these optimum conditions are determined by multiple laboratory- or pilot-scale experiments. A significant effort has been previously conducted and published in peer reviewed literature by various researchers; however, variability in their experimental conditions does not conclude to universal operating conditions for landfill leachate. To determine a generalized Fenton operating condition for landfill leachate, experiments were conducted with respect to COD loading factor (L_{COD}). L_{COD} is defined as the COD with respect to available oxygen obtained through the use of hydrogen peroxide.
The experiments were conducted at pH 3.5 and ratio of H_2O_2/Fe^{2+} as 1.8. These pH and ratio of Fenton reagent doses were selected based on optimum conditions obtained in published peer reviewed literature for leachate treatment. The experiments were planned to be conducted at four COD loading condition at L_{COD} setting at 0.25, 0.5, 0.75 and 1.0 at three initial COD conditions. The oxidation efficiency and biodegradability improvement were evaluated after Fenton treatment of leachates.

Leachate Characterization

Leachate samples were collected from Florida class-I landfill located in Palm Beach County, Florida. The samples were kept in the dark at 4 ^oC and were brought to room temperature (23±1 ^oC) prior to experiments. The leachate samples were characterized and leachate quality parameters were determined as shown in Table 1.

Table 1: Leachate quality parameters

Parameter	Unit	Average	Std dev.
pH		7.81	
DO	mg/L	2.64	
Temp	^{o}C	20.5	
Conductivity	mScm-1	10. 6	
TDS	mg/L	7057.1	243.4
Alkalintiy	mg $CaCO_3$/L	2385	35.4
COD	mg/L	1370	56.9
TOC	mg/L	259.9	8.0

Experimental Procedure

The Fenton experiments were performed in a bench scale jar testing apparatus. The flowchart of experimental procedure is shown in Figure 1. Leachate samples were taken in a 2 L beaker and pH was adjusted to 3.5 using 10 M H_2SO_4 and 10 M NaOH solutions. Amount of H_2SO_4 and NaOH added were recorded, whenever pH was adjusted. The solution mixing at 100 rpm was started and previously dissolved (in 10 mL DI) desired amount of $FeSO_4 \cdot 7H_2O$ was added in the solution. Approximately 1 minute after the $FeSO_4 \cdot 7H_2O$ addition desired amount of H_2O_2 solution was added in single step. Treated leachate samples (20 mL) were collected at 0.5, 1, 5, and 10 minutes in Erlenmeyer flasks while continue mixing the solution at 100 rpm. The pH of solution were monitored and measured at the end of each time interval. After 10 minutes of mixing at 100 rpm, the solution pH was adjusted to 7.0 by adding 10 M NaOH for coagulation process. The mixing speed was kept constant at 100 rpm. Coagulation was conducted for 5 min (i.e. from 10 minutes to 15 minutes from the start of experiment) and sample was collected. After coagulation, solution mixing speed was reduced to 30 rpm for flocculation for 15 minutes. After flocculation, mixing was stopped to precipitate all the coagulated-flocculated organic matter. 1 hr 15 minutes later from the start of experiment (or 45 minutes after stopping the mixing) the samples of supernatant and sediments were collected. Right after collecting each sample in a flask, pH of samples were adjusted to >12 by adding 2 mL of 10 M NaOH solution to stop the oxidation activity. Each sample was analyzed for TOC and BOD. The TOC values were converted to COD values based on a relationship obtained through various COD and TOC analysis. Experiments with initial conditions as shown in Table 2 were conducted in third quarter.

Table 2: Experiment conditions

pH		3.50	3.50	3.50	3.50
L_{COD}		0.25	0.50	0.75	1.00
COD_0	mg/L	171.25	342.50	685.00	1370.00
H_2O_2/Fe^{2+} (w/w)		1.80	1.80	1.80	1.80
H_2O_2 required	mg/L	1457.45	1457.45	1943.26	2914.89
Fe^{2+} required	mg	809.69	809.69	1079.59	1619.39

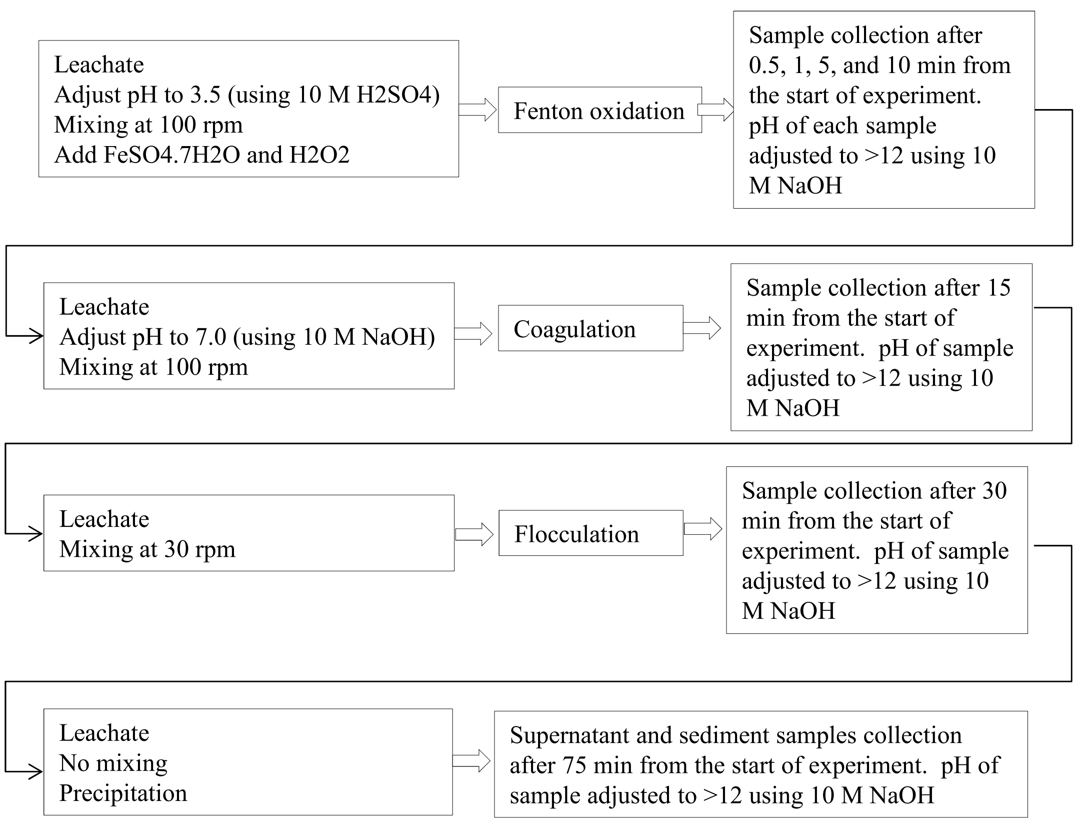

Figure 1: Flowchart of Fenton process experimental procedure

Experimental Results

Relationship between TOC and COD of landfill leachate

From the preliminary experiments it was observed that the applied COD digestion method (HACH method) was not able to sufficiently oxidize organic matter present in leachate and

HACH method of COD analysis was not providing confident COD results. Therefore, leachate samples were analyzed for TOC and COD at various dilutions as shown in Table 3 and a relationship between COD and TOC was established. COD and TOC of leachate had a relationship of COD=6.27*TOC-16.87 with a coefficient of determination 0.997 as shown in Figure 2. Afterwards all the experimental samples were analyzed for TOC and corresponding COD values were reported.

Table 3: TOC and COD of leachate samples

Leachate dilution	**TOC mg/L**	**COD mg/L**
1:100	3.91	8.33
1:50	6.72	24.33
1:40	7.38	29.00
1:25	9.96	46.33

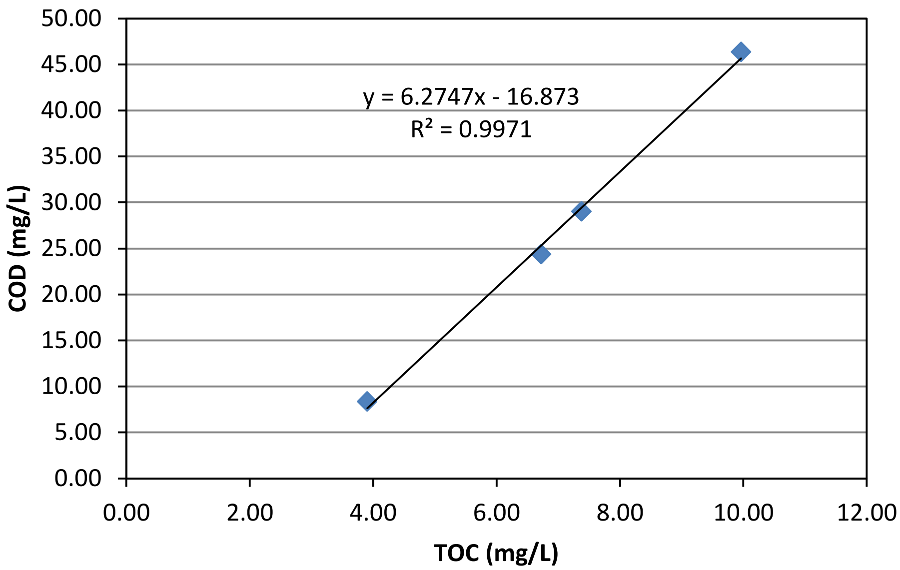

Figure 2: Relationship between leachate TOC and COD

Effect of COD loading on leachate treatment efficiency

Oxidation efficiency varied in the range of 0.15 to 0.48 for L_{COD} in than range of 0.25 to 1.0 as shown in Table 4. The maximum oxidation efficiency was observed at L_{COD} of 0.75. A maximum of 48% COD was removed by Fenton oxidation at L_{COD} 0.75 and 60% COD was removed by Fenton Process.

Table 4: Effect of COD loading on oxidation efficiency, Percentage COD removal by Fenton oxidation and Fenton process

COD_0	Concentration of H_2O_2 used	L_{COD}	Ox. Eff.	COD removal by oxidation	COD removal by Fenton process
mg/L	mg/L			%	%
171.25	1457.45	0.25	0.15	27.71	35.89
342.50	1457.45	0.50	0.39	63.00	68.82
685.00	1943.26	0.75	0.48	47.92	59.90
1370.00	2914.89	1.00	0.44	39.30	57.88

The efficiency of H_2O_2 for organic matter removal also observed to be increased with the increase in L_{COD} from 0.25 to 0.75 as shown in Table 5. The experimental results also show that organic matter removal was primarily observed due to Fenton oxidation as compared to Fenton process. Additional experiments are underway for L_{COD} in the range of 0.15 to 1.0 for various initial COD values.

Table 5: Effect of COD loading on COD removal per H_2O_2 by Fenton oxidation and Fenton process

COD_0	Concentration of H_2O_2 used	L_{COD}	COD removed/H_2O_2	
mg/L	mg/L		Oxidation	Fenton process
171.25	1457.45	0.25	0.07	0.09
342.50	1457.45	0.50	0.18	0.20
685.00	1943.26	0.75	0.22	0.28
1370.00	2914.89	1.00	0.21	0.31

Fenton oxidation showed a significant improvement in leachate biodegradability as represented by increase in BOD after Fenton treatment as shown in Table 6.

Table 6: Effect of COD loading on BOD of leachate after Fenton oxidation and Fenton process treatment

COD_0	Concentration of H_2O_2 used	L_{COD}	BOD_0	BOD	BOD
mg/L	mg/L		mg/L	oxidation	Fenton process
171.25	1457.45	0.25			
342.50	1457.45	0.50	60.86	69.57	50.41
685.00	1943.26	0.75	70.44	74.36	63.31
1370.00	2914.89	1.00	44.10	102.63	79.51

Typical photos of the experiment results are shown in appendix A-1 during Fenton oxidation of leachate and A-2 after Fenton oxidation of leachate.

CONTINUED WORK: QUARTER FOUR

Additional experiments are underway to determine the COD removal efficiencies at COD loading in the range of 0.25 to 1.0 at different initial COD concentrations. The upcoming results and the results reported in this report should establish a generalized Fenton treatment conditions for landfill leachate. The results will also be evaluated for biodegradability improvement and the cost estimation will be conducted for the Fenton Process for leachate treatment.

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Appendix A

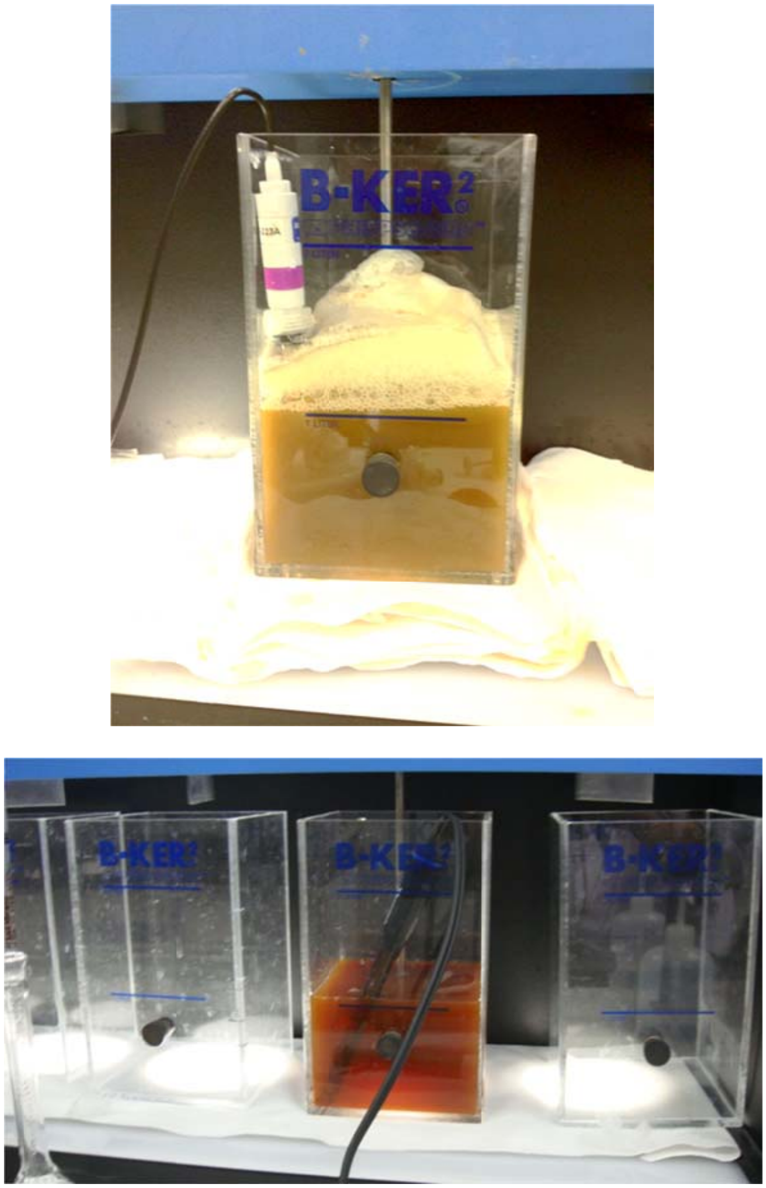

Figure A-1: During Fenton oxidation of leachate

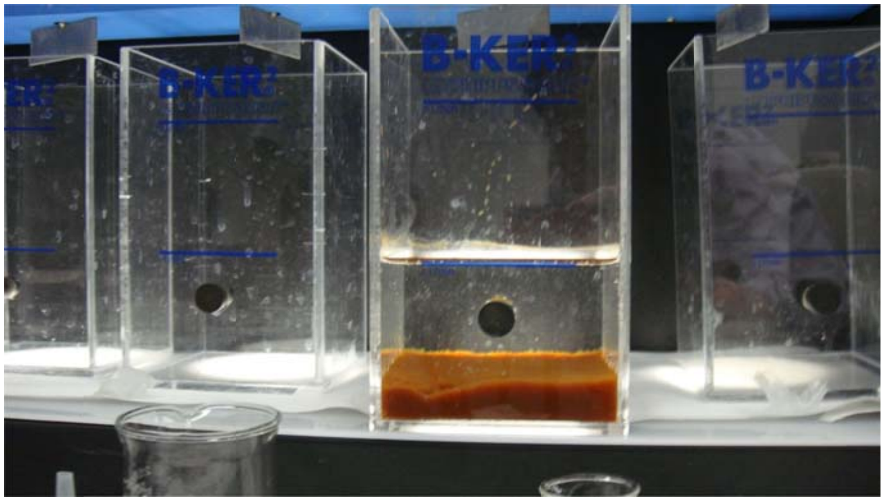

Figure A-2: After Fenton oxidation of leachate

Appendix B

Raw Experimental Data

H_2O_2 solution characteristics

Density	1.135	g/mL
Solution strength	35.00	% (w/w)
	0.39725	g/mL
	397.25	mg/mL
Fe^{2+} from $FeSO_4{\cdot}7H_2O$		
MW $FeSO_4{\cdot}7H_2O$	278.02	
MW Fe^{2+}	56.00	
$Fe^{2+}/FeSO_4.7H_2O$	0.20	

Expriment #		11	12	13	14	15	16	17	18	19	**20**	**21**
pH		**2.00**	**2.00**	**2.00**	**3.50**	**3.50**	**3.50**	**6.00**	**6.00**	**6.00**	**3.50**	**3.50**
Lcod		**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**0.25**	**1.00**
Leacahte dilution		**1:4**	**1:2**	**1:1**	**1:4**	**1:2**	**1:1**	**1:4**	**1:2**	**1:1**	**1:8**	**1:1**
COD_0	mg/L	342.50	685.00	1370.00	342.50	685.00	1370.00	342.50	685.00	1370.00	171.25	1370.00
H_2O_2/Fe^{2+} (w/w)		1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80
H_2O_2 required	mg/L	1,457.45	1,943.26	2,914.89	1,457.45	1,943.26	2,914.89	1,457.45	1,943.26	2,914.89	1,457.45	2,914.89
Fe2+ required	mg	809.69	1,079.59	1,619.39	809.69	1,079.59	1,619.39	809.69	1,079.59	1,619.39	809.69	1,619.39
Amount of H2O2 solution	mL/L	3.67	4.89	7.34	3.67	4.89	7.34	3.67	4.89	7.34	3.67	7.34
Amount of FeSO4.7H2O salt	mg	4,019.83	5,359.78	8,039.67	4,019.83	5,359.78	8,039.67	4,019.83	5,359.78	8,039.67	4,019.83	8,039.67
Sampling time		**TOC**										
min												
0.00	raw	45.87	91.17	236.30	69.43	148.01	248.62	85.11	93.77		61.42	259.91
0.50	oxidation	56.93	70.49	151.67	38.85	93.19	149.36	112.23	69.05		41.55	194.02
1.00	oxidation	22.71	72.61	151.76	26.64	106.85	152.05	51.25	58.18		51.28	174.55
5.00	oxidation	22.09	70.39	126.66	27.38	78.38	151.95	45.10	71.64		45.15	170.84
10.00	oxidation	38.65	60.01	117.62	26.95	70.39	146.86	20.60	94.05		49.01	166.97
15.00	coagulation	37.31	61.55	137.05	72.13	80.49	130.89	17.04	91.36		39.14	157.04
30.00	flocculation	37.21	30.16	118.39	54.81	63.66	131.18	46.35	77.61		44.65	153.39
75	supernatent	7.70	28.69	86.65	23.50	60.97	106.27	5.71	26.83		40.34	119.85
75.00	Sediment	111.56	135.22	209.66	148.49	110.31	366.53	133.97	95.79		105.02	314.19

Processes

pH		**2.00**	**2.00**	**2.00**	**3.50**	**3.50**	**3.50**	**6.00**	**6.00**	**6.00**	**3.50**	**3.50**
Lcod		**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**0.25**	**1.00**
Raw leachate		45.87	91.17	236.30	69.43	148.01	248.62	85.11	93.77	0.00	61.42	259.91

Oxidation after 5 min		22.09	70.39	126.66	27.38	78.38	151.95	45.10	71.64	0.00	45.15	170.84
Coagulation		37.31	61.55	137.05	72.13	80.49	130.89	17.04	91.36	0.00	39.14	157.04
Flocculation		37.21	30.16	118.39	54.81	63.66	131.18	46.35	77.61	0.00	44.65	153.39
Supernatent		7.70	28.69	86.65	23.50	60.97	106.27	5.71	26.83	0.00	40.34	119.85
Sediment		111.56	135.22	209.66	148.49	110.31	366.53	133.97	95.79	0.00	105.02	314.19
NET TOC removed by Fenton process		38.16	62.48	149.66	45.94	87.04	142.35	79.40	66.93	0.00	21.08	140.06

TOC removed

Oxidation		23.78	20.77	109.65	42.05	69.63	96.66	40.01	22.12	0.00	16.27	89.07
Coagulaiton		-15.22	8.85	-10.39	-44.74	-2.12	21.06	28.06	-19.72	0.00	6.01	13.79
Flocculation		0.10	31.38	18.66	17.31	16.83	-0.29	-29.31	13.75	0.00	-5.51	3.65
Precipitation		29.51	1.47	31.74	31.32	2.69	24.91	40.64	50.77	0.00	4.31	33.54
NET TOC removed		38.16	62.48	149.66	45.94	87.04	142.35	79.40	66.93	0.00	21.08	140.06

COD

0.00	raw	270.93	555.18	1465.87	418.79	911.85	1543.11	517.16	571.48	-16.87	368.53	1614.00
0.50	oxidation	340.34	425.43	934.79	226.88	567.86	920.30	687.35	416.38	-16.87	243.82	1200.55
1.00	oxidation	125.61	438.71	935.39	150.30	653.55	937.20	304.73	348.18	-16.87	304.92	1078.41
5.00	oxidation	121.75	424.83	777.88	154.94	474.92	936.60	266.11	432.67	-16.87	266.41	1055.08
10.00	oxidation	225.67	359.65	721.15	152.23	424.83	904.61	112.40	573.29	-16.87	290.63	1030.80
15.00	coagulation	217.22	369.31	843.05	435.69	488.20	804.43	90.07	556.39	-16.87	228.71	968.53
30.00	flocculation	216.62	172.38	725.97	327.06	382.58	806.24	273.95	470.09	-16.87	263.31	945.60
75	supernatent	31.47	163.15	526.82	130.56	365.68	649.93	18.97	151.50	-16.87	236.26	735.17
75.00	Sediment	683.13	831.59	1298.70	914.87	675.28	2283.01	823.74	584.15	-16.87	642.09	1954.60
Lcod calculated		0.40	0.61	1.07	0.61	1.00	1.13	0.75	0.63	(0.01)	0.54	1.18
	Lcod	0.50	0.75	1.00	0.50	0.75	1.00	0.50	0.75	1.00	0.25	1.00
	OxEff	0.22	0.14	0.50	0.39	0.48	0.44	0.37	0.15	-	0.15	0.41

COD at each Process

pH		**2.00**	**2.00**	**2.00**	**3.50**	**3.50**	**3.50**	**6.00**	**6.00**	**6.00**	**6.00**	**6.00**
Lcod		**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**0.50**	**0.75**	**1.00**	**1.00**	**1.00**
Raw leachate		270.93	555.18	1465.87	418.79	911.85	1543.11	517.16	571.48	-16.87	368.53	1614.00
Oxidation after 5 min		121.75	424.83	777.88	154.94	474.92	936.60	266.11	432.67	-16.87	266.41	1055.08
Coagulation		217.22	369.31	843.05	435.69	488.20	804.43	90.07	556.39	-16.87	228.71	968.53
Flocculation		216.62	172.38	725.97	327.06	382.58	806.24	273.95	470.09	-16.87	263.31	945.60
Supernatent		31.47	163.15	526.82	130.56	365.68	649.93	18.97	151.50	-16.87	236.26	735.17
Sediment		683.13	831.59	1298.70	914.87	675.28	2283.01	823.74	584.15	-16.87	642.09	1954.60
NET COD removed by Fenton process		239.47	392.03	939.05	288.23	546.17	893.18	498.19	419.98	0.00	132.26	878.83

COD removed

Oxidation		149.19	130.36	687.99	263.85	436.93	606.52	251.06	138.81	0.00	102.11	558.91
Coagulaiton		-95.47	55.52	-65.18	-280.75	-13.28	132.17	176.04	-123.72	0.00	37.70	86.55
Flocculation		0.60	196.92	117.08	108.63	105.61	-1.81	-183.89	86.30	0.00	-34.60	22.93
Precipitation		185.15	9.23	199.16	196.50	16.90	156.31	254.98	318.59	0.00	27.05	210.43
NET COD removed		239.47	392.03	939.05	288.23	546.17	893.18	498.19	419.98	0.00	132.26	878.83

	Oxidation efficiency vs L_{COD}		
	pH		
LCOD	**2**	**3.5**	**6**
0.25		0.15	
0.5	0.22	0.39	0.37
0.75	0.14	0.48	0.15
1	0.50	0.44	

% COD removed

Fenton Oxidation		55.06	23.48	46.93	63.00	47.92	39.30	48.54	24.29	0	27.708781	34.62919
Fenton Process		88.39	70.61	64.06	68.82	59.90	57.88	96.33	73.49	0	35.889643	54.45054

Amount of COD removed per mg H_2O_2

Fenton Oxidation		0.10	0.07	0.24	0.18	0.22	0.21	0.17	0.07	-	0.07	0.19
Fenton Process		0.16	0.20	0.32	0.20	0.28	0.31	0.34	0.22	-	0.09	0.30

Oxidation efficiency

Fenton Oxidation		0.22	0.14	0.50	0.39	0.48	0.44	0.37	0.15	-	0.15	0.41
Fenton Process		0.35	0.43	0.69	0.42	0.60	0.65	0.73	0.46	-	0.19	0.64

Sampling time		**BOD**										
min												
0.00	raw	14.04	17.96	22.88	60.86	70.44	44.10	22.27	6.72	46.15		
0.50	oxidation	24.82	30.18	53.44	67.11	78.08	77.19	31.91	39.88	58.58		
1.00	oxidation	22.75	39.66	61.51	69.32	86.06	78.02	32.01	32.76	62.31		
5.00	oxidation	22.25	42.02	67.74	69.57	74.36	102.63	22.08	43.95	74.14		
10.00	oxidation	14.43	48.56	72.66	69.27	80.51	100.09	22.21	42.85	85.47		
15.00	coagulation	26.06	42.07	84.95	71.03	74.14	102.98	29.74	42.65	89.75		
30.00	flocculation	37.00	39.82	83.44	72.22	74.48	97.78	30.82	53.24	96.52		
75	supernatent	17.17	38.75	77.94	50.41	63.31	79.51	14.91	17.58	-24.96		
75.00	Sediment	47.72	58.12	115.85	56.19	61.42	144.27	26.44	48.67	84.66		

BOD improved

Oxidation		8.21	24.06	44.86	8.71	3.92	58.53	-0.19	37.22	27.99
Coagulaiton		3.81	0.06	17.22	1.46	-0.22	0.35	7.66	-1.29	15.62
Flocculation		10.93	-2.25	-1.52	1.19	0.34	-5.21	1.08	10.59	6.76
Precipitation		-19.83	-1.07	-5.49	-21.81	-11.16	-18.27	-15.91	-35.67	-121.48

Appendix C

Submitted Manuscript

Title: Statistical Analysis of Optimum Fenton Oxidation Conditions for Landfill Leachate Treatment

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Abstract

Optimal operating conditions as observed by peer reviewed publications for Fenton oxidation of raw and biological and coagulation treated leachates were reviewed and statistically analyzed. For the first-stage Fenton oxidation, the optimal pH range of 2.5 to 4.5 was observed for raw and coagulation treated leachates with a median pH of 3.0, whereas, for biologically treated leachate the optimum pH range was 2.5 to 6.0 with a median pH of 4.25. Theoretically, the optimal ratio of H_2O_2/Fe^{2+} should be the ratio of rate constants of the reactions between $OH^{\bullet}$ radical with Fe^{2+} and H_2O_2, which is approximately 11; however, for leachate treatment, a median optimum relative dose of 1.8 (w/w) (molar ratio: 3.0) was observed. Biologically treated leachate showed relatively lower optimum ratio of H_2O_2/Fe^{2+} doses (median: 0.9 w/w) as compared to raw (median: 2.4 w/w) and coagulation treated (median: 2.8 w/w) leachate. Median absolute doses of H_2O_2 and Fe^{2+} were 1.2 mg H_2O_2/mg of initial COD (COD_0) and 0.9 mg Fe^{2+}/COD_0, respectively and raw leachate required higher reagent doses as compared to pretreated leachates. A universal Fenton oxidation relationship between oxidation efficiency (η) and COD loading factor (L_{COD}) for landfill leachate treatment was developed. As L_{COD} increases from 0.0.03 to 72.0, η varies linearly as $\eta = 0.733\,L_{COD} - 0.182$. This robust linear relationship between L_{COD} *and* η holds for Fenton oxidation of raw as well as biological and coagulation treated leachates. The relationship was validated using Leave-one-out cross validation technique and the errors in predicting η by using L_{COD} were evaluated by applying Monte Carlo Simulation. As a result, the relationship can be used as a universal equation to predict Fenton oxidation efficiency for a given COD_0 loading in the range of 0.0.3 to 72.0 for landfill leachate treatment.