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Activated Carbon Testing for the 200 Area Effluent Treatment Facility

R. N. Wagner

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Abstract: This report documents pilot and laboratory scale testing of activated carbon for use in the 200 Area Effluent Treatment Facility peroxide decomposer columns. Recommendations are made concerning column operating conditions and hardware design, the optimum type of carbon for use in the plant, and possible further studies.

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**ACTIVATED CARBON TESTING
FOR THE
200 AREA EFFLUENT TREATMENT FACILITY**

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Rust Federal Services of Hanford, Inc.

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January 1997

EXECUTIVE SUMMARY

Three grades of activated carbon, produced by two different manufacturers, were performance tested for the 200 Area Effluent Treatment Facility (ETF) hydrogen peroxide decomposer columns during October and November, 1996. Two of the carbon grades (mesh sizes 8x30 and 12x40) are manufactured from bituminous coal by the Calgon Carbon Corporation under the brand name Centaur. The third carbon type (mesh size 8x20) is produced from coconut shell by Cameron Yakima, Inc.

Each carbon was subjected to four separate tests: (1) washing conditions required for fines removal; (2) bed expansion under upflow; (3) kinetics studies for a comparison of peroxide destruction potential; and (4) pilot scale peroxide destruction efficiency. Tests 1, 2, and 4 were conducted on a pilot scale in a six-inch diameter clear plastic test column, while Test 3 was conducted on a laboratory scale in beakers.

Test 1 showed that removal of carbon fines from a new bed is best accomplished at a superficial velocity greater than the ETF decomposer column maximum design flow of 12.0 gpm/ft² (equivalent to a flowrate of 85 gpm through each column). Optimum fines removal, with minimal loss of carbon particles in the desirable mesh size range, occurred around 17 to 20 gpm/ft² (equivalent to 120 to 140 gpm through each decomposer column). Also, both of the bituminous coal carbons (Calgon Centaur brand) appeared to wash more cleanly than the coconut-based carbon (Cameron brand). After washing was completed and the flow was reduced to 12 gpm/ft² to simulate design process conditions, the void space above the beds of both Centaur carbons was completely free of visible particulates. With the Cameron carbon, even after both washing and the upflow testing of Test 2, the water above the bed remained slightly turbid, and a few carbon particles continued to be lofted above the bed at the design superficial velocity of 12 gpm/ft². Based on visual observations of wetted carbon, the coconut-based material from

Cameron appeared somewhat muddy and seemed to contain a significantly broader range of particle sizes than either of the Centaur carbons

Test 2 was performed to gather bed expansion data for each carbon. The maximum superficial velocity tested was 25.5 gpm/ft² for each carbon. As expected, maximum bed expansion was greatest with the finest mesh carbon, Centaur 12x40, at 122%. However, the Cameron 8x20 mesh carbon bed, at 81%, exhibited about double the maximum expansion of the Centaur 8x30 mesh, at 42%. This was unexpected, given that the densities of both carbons are similar, and that the Centaur 8x30 should contain a finer average mesh of carbon than the Cameron 8x20.

Test 3, which was conducted on a laboratory scale in beakers, clearly showed that bituminous coal-based carbon is superior to coconut-based carbon for peroxide destruction. Centaur 12x40 is the best choice for peroxide destruction, with a calculated peroxide destruction reaction rate nearly double that of Centaur 8x30 and over 2.5 times that of Cameron 8x20. The performance difference between the two Calgon Centaur carbons is most likely due to the greater surface area of the 12x40 mesh. But even if an allowance is made for the coarser mesh size rating of Cameron 8x20, its relatively poor performance is most likely due to the lower catalytic activity of coconut shell.

Test 4 was conducted primarily to confirm the peroxide destruction efficiency results of Test 3 in the pilot scale column, under upflow at the maximum design superficial velocity of the ETF decomposer columns. Both Calgon Centaur carbons were superior to the Cameron carbon, as expected. However, Centaur 8x30 and Centaur 12x40 appeared nearly equal in peroxide destruction performance under the pilot scale conditions. It is possible that other factors beyond the control or scope of this test, such as varying carbon bed flow regimes or the effect of limited bed residence time, may have come into play during pilot scale testing. Nevertheless, the relatively strong performance of Centaur 12x40 in the kinetics studies of Test 3 indicates that it is most likely the best choice for the ETF decomposer columns.

Visual observations during Test 4 indicate that most of the carbon bed loss from the ETF decomposer during processing may be caused by lofting of carbon particles on oxygen gas bubbles formed within the bed. These bubbles, which are produced during the carbon-catalyzed destruction of peroxide, become attached to carbon particles in the bed and provide sufficient buoyancy to loft the particles up to the top of the column, where they become entrained in the effluent stream. It was noted that if the lofted particles are given an opportunity to contact a vapor space (i.e., gas/liquid interface), the attached bubbles are released almost immediately, and the particles sink fairly rapidly back down to the bed due to the relatively high true density of fully-wetted carbon (2.3 g/cc).

The following recommendations are based on the results of this study:

- Washing to remove fines from a new carbon bed should be performed at greater than the 85 gpm maximum design process flowrate of the decomposer columns. Test results indicate that a flow of 120 to 140 gpm through each column, maintained for about one hour, should provide optimal fines removal for either of the bituminous coal-based Centaur carbons.
- A bituminous coal-based product should be the activated carbon of choice for the ETF peroxide decomposer columns. Among the three carbons tested, Centaur 12x40 should provide the greatest degree of peroxide destruction. It is the recommended carbon to maximize protection of downstream components, such as reverse osmosis membranes, from the effects of residual hydrogen peroxide.
- The ETF peroxide decomposer internal design, specifically the discharge header configuration, should be examined for improvements to counteract the gas bubble buoyancy effect on carbon particles. Providing an opportunity for lofted carbon particles to degas (lose their buoyant bubbles) at a gas/liquid interface would also help reduce the quantity of carbon lost from the bed. Also, enclosing the discharge header in some type of

fine mesh sheath or screen would reduce the quantity of carbon that is pulled into the header, and may encourage lofted particles to contact one another at the screen surface so that their attached gas bubbles can combine and be released from the carbon.

- The use of unnecessary excess hydrogen peroxide in the ETF ultraviolet oxidation process should be avoided. This will minimize the concentration of peroxide that must be destroyed in the decomposer columns. Less peroxide will produce fewer gas bubbles, hence, fewer carbon particles will be lofted to the decomposer column discharge headers.
- The existence and effectiveness of metallic or ceramic catalysts for peroxide destruction should be investigated. Such catalysts, if they exist and are practical for use in the ETF peroxide decomposer columns, would have the advantage of negligible fines production and potentially long life because of their material characteristics and ability to withstand vigorous chemical cleaning. Extended catalyst life would provide operational cost savings by substantially reducing the amount of solid waste generated from the decomposer columns over the life of the plant. Also, the greater density of a metal or ceramic catalyst would likely eliminate the gas bubble buoyancy effect.

Approximately 2.5 hours of VHS format videotape was recorded during the actual testing. This video footage is available for viewing upon request.

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- R. L. Loper - Technician support for performance of testing.

Their support contributed greatly to the value of this testing.

TRADEMARK INFORMATION

The following commercial products are named in this test report:

- Centaur brand bituminous coal based activated carbon, mesh grades 8x30 and 12x40, a product of Calgon Carbon Corporation, Burlingame, CA, USA.
- Cameron brand coconut shell based activated carbon, mesh grade 8x20, a product of Cameron Yakima, Inc., Yakima, WA, USA.
- CHEMetrics brand colorimetric test kits for hydrogen peroxide concentration, kits K-5510, K-5510B, and K-5510D, products of CHEMetrics, Inc., Calverton, VA, USA.

This report is based solely upon the technical phenomena observed during laboratory scale and pilot scale testing, and does not represent any type of endorsement regarding the products listed above by either the U.S. Department of Energy or any of its contractors or subcontractors.

ACTIVATED CARBON TESTING FOR THE 200 AREA EFFLUENT TREATMENT FACILITY

1.0 INTRODUCTION

1.1 Test Objective

The objective of this test was to obtain performance data on activated carbon to support selection of the optimum carbon catalyst for use in the 200 Area Effluent Treatment Facility (ETF) peroxide decomposer columns. Data was needed in the following areas:

- (a) Optimum conditions for washing fines out of new carbon
- (b) Degree of carbon bed expansion during upflow operation over the range of ETF process flow rates
- (c) Relative efficiency for destruction of excess hydrogen peroxide in the ETF process stream.

1.2 Equipment Background

The ETF uses activated carbon beds in 3-ft diameter by 15-ft tall vessels to decompose excess hydrogen peroxide (H_2O_2) from the ultraviolet light oxidation (UV/OX) process used for organic destruction. Each bed nominally contains 1200 lbs of carbon, which fills the vessel up to the 6-ft level. The decomposer operates in an upflow configuration at a maximum design flow rate of 85 gpm. Based on the column diameter, the resultant superficial velocity at this flow rate is 12 gpm/ft². Process water temperature ranges from 68° F to 140° F. When the ETF is treating 242-A Evaporator process condensate, the pH is held at 4 by a pH adjustment tank immediately upstream of the decomposer. The pH will range between 5 and 6 when the ETF begins treating 200-UP-1 groundwater in calendar year 1997.

The upflow configuration of the decomposer columns is necessary for removal of gas bubbles generated during the catalytic destruction of peroxide. It was known that the carbon beds fluidize and expand under this upflow, but the degree of expansion and fluidization was unknown because it is not possible to directly observe the decomposer beds in the ETF under process conditions.

1.3 Problem Description

Problems that have been experienced with the ETF peroxide decomposers fall into two categories:

- **High Equipment Differential Pressures** - ETF process operations have been hampered by high differential pressure (dP) across the peroxide decomposers, and across the fine

filter (0.5 μm nominal) located immediately downstream of the decomposers. The high dP appears due to carbon particles, possibly fines from inadequate bed washing, that lodge in the decomposer discharge headers and blind the fine filter. Repeated attempts to wash out the fines did not remedy the situation. The ETF main treatment train (MTT) has been shut down repeatedly to backwash the decomposers and fine filter, and for chemical cleaning of the fine filter. High dP conditions have generally returned within hours of restarting the MTT.

- **Inadequate Peroxide Destruction Efficiency** - The decomposers have exhibited reduced peroxide destruction efficiency with coconut shell-based carbon. The decomposers were designed to reduce a 200 ppm worst-case inlet peroxide concentration to 0.5 ppm at the outlet, when running at the 85 gpm design maximum flow rate. The nominal inlet concentration ranges from 30 to 50 ppm. With the present coconut shell carbon, the decomposer has only been able to consistently reduce this nominal inlet concentration to 5-10 ppm at the outlet. Manufacturers of bituminous coal-based catalytic carbon maintain that their product should destroy peroxide at about ten times the rate of coconut shell carbon. Hence, a change to coal-based carbon for the decomposers has been contemplated.

2.0 TYPES OF CARBON TESTED

The following types of activated carbon were the subject of this testing:

- (a) Calgon Carbon Corporation Centaur 12x40 mesh (bituminous coal-based)
- (b) Calgon Carbon Corporation Centaur 8x30 mesh (bituminous coal-based)
- (c) Cameron Yakima, Inc. 8x20 mesh (coconut shell-based, installed in the ETF decomposer columns during summer 1996).

3.0 TEST DESCRIPTION AND EQUIPMENT

Each carbon was subjected to four separate tests: (1) washing conditions required for fines removal; (2) bed expansion under upflow; (3) kinetics and isotherm studies for peroxide destruction; and (4) peroxide destruction efficiency. Tests 1, 2, and 4 were conducted in a 6-inch diameter clear plastic test column, while Test 3 was conducted on a laboratory scale.

The equipment and procedures for the four tests are described in Appendix A. A schematic of the test skid is shown in Figure 1.

Figure 1: Test Skid Schematic for Carbon Tests 1, 2, and 4

4.0 DATA AND RESULTS

4.1 TEST 1: Time Required to Wash Fines from New Carbon

The test procedure is described in detail in Appendix A. The test column was loaded with a 12-inch depth bed of pre-soaked carbon. Beginning at zero flow, upflow through the column was increased incrementally to a maximum of 5-6 gpm. Tables 1 through 3 display the raw data taken during this test. Note that “elapsed time” in the first column of Tables 1 through 3 refers to the time at which the flow rate was increased to the next higher increment. For example, referring to Table 1 for the testing of Calgon Centaur 12x40 carbon, at 11 minutes elapsed time, the flow rate was increased from 1.0 to 1.5 gpm, and at 18 minutes, the flow was increased again to 2.0 gpm. Thus, the flow was held at 1.0 gpm for a total of 7 minutes.

Column effluent samples were taken from Sample Port 2 (see Figure 1) approximately 2 to 2.5 minutes into the elapsed time for each incremental flow rate. Samples of approximately 500 mL were drawn into a 1-liter container and well mixed. A 25-mL aliquot was then analytically transferred to a tared weighing pan. Each pan was dried overnight in a 110 °C hot air oven. The following day, the weighing pans were retrieved from the oven, cooled, and weighed on a top-loading Mettler pan balance. Table 4 presents a summary of the solids, or fines washout, data from Tables 1 through 3. The column in Table 4 labeled “% of Total” provides a rough indication of the fraction of total fines that was washed out of each bed at each flow rate.

Fines washout data is also presented graphically for each carbon type in Figures 2 through 4.

Note that the initial “slug” of fines released from the Cameron 8x20 carbon was about an order of magnitude greater than the initial fines released from either of the Calgon products. An additional observation not specifically recorded in the tables was that both of the Calgon carbons appeared more robust and uniform in grain size than the Cameron carbon.

Turbidity instrumentation was not available during the first fines washout run, in which the Calgon Centaur 12x40 was tested. However, the solids levels present in the column effluent samples provide adequate data for comparison between the three carbons. The release of more fines from Centaur 12x40 than from Centaur 8x30 is readily explained by the finer overall mesh size of the 12x40 product. However, relative mesh size does not explain the large quantity of fines released from Cameron 8x20 compared to either of the Calgon products. Given that the relative densities of all three carbons are approximately equal, the primary reason for the large quantity of fines present in the Cameron coconut shell carbon appears to be that it is more prone to particle breakup due to normal handling and operations, possible because it is a cellulose based product and has a lower degree of hardness than the Calgon bituminous coal based material. All three carbons were handled with equal care during the carbon preparation step, and the sack containing the Cameron product gave no appearance of abuse.

The column effluent pH for Cameron carbon also was the highest of the three carbons, and was the only carbon to show an initial effluent pH above 8.0. This may have been caused by the alkalinity of residual ash present in the carbon, which in turn may be due to the coconut shell origin of Cameron carbon.

This testing indicates that the optimum superficial velocity for fines washing is greater than the design flow of 2.4 gpm/ft² because a large quantity of fines was released from the three carbon at the higher flow rates. However, the maximum beneficial superficial velocity for fines removal appears to be around 20 gpm/ft². Above this flow rate, a significant quantity of discrete carbon particles within the rated mesh size becomes entrained with the column effluent, leading to an undesirable loss of bed carbon. Further, above 20 gpm/ft², bed expansion becomes extreme, particularly for Calgon Centaur 12x40 because of its smaller mesh size. In a column with a smaller ratio of height to diameter, or for a deeper bed in relation to the vessel height, bed expansion would exceed the available column height.

The degree to which fines removal was successful is shown by the clarity of the water above the carbon bed when the flow rate was dropped to the design superficial velocity at the end of each test run. The clarity of the water above both Calgon carbon beds was outstanding after fines removal.

Table 1: Fines Flushing Data for Calgon Centaur 12x40 Carbon

Elapsed Time [min]	Flow Rate [gpm]	Water Temp [°F]	pH	Bed Depth [in]	Column dP [psi]	Turbidity [NTU]	Effluent Solids [ppm]	Comments/Observations
0	0	72		12	0	Meter not available		A layer of grey-black scum is visible on top of stagnant water in column. Some air bubbles entrained in the bed.
11	1.0	72	5.95	13.6	0		300	Water above carbon was very dark initially; cleared up after about 4.5 minutes.
18	1.5	72.5	6.8	13.8	0			Discrete coarse carbon particles visible above bed, appear to be coarse fines.
28	2.0	72.5	6.34	16	2		112	Fines continuing to come off bed.
44	2.5	69	6.5	17.3	2.5		164	As above, with some small bed particles entrained in upflow.
56	3.0	65.5	6.49	19.2	3.5		120	Increased number of bed particles entrained in upflow. Fines continuing to wash off.
66	3.5	63.5		21.5	4.75			As above.
82	4.0	62.5	6.29	24	5.75		92	Increasing number of bed particles lofting out of column with fines. Fines in effluent sample settle quickly.
90	4.5	62	6.6	~27	6.9		100	Top of bed becoming ill-defined due to turbulence at interface. Otherwise, as above.
97	5.0	62	6.65	~31	8.0			Large number of carbon particles exiting column. Very few fines are visible. Flow rate has exceeded that necessary for effective fines removal.
≥120	2.5	65		17.6				Bed now at ~150% expansion for design flow rate. Water above bed is very clear, free of visible fines.

Table 2: Fines Flushing Data for Calgon Centaur 8x30 Carbon

Elapsed Time [min]	Flow Rate [gpm]	Water Temp [°F]	pH	Bed Depth [in]	Column dP [psi]	Turbidity [NTU]	Effluent Solids [ppm]	Comments/Observations
0	0	71		12.5	0			Grey-black scum on top of stagnant water in column, but less than for Centaur 12x40. Carbon grains are noticeably coarser, as expected for mesh size.
16	1.0	72.5	7.5	12.5	1	38.1/2.0	188	Effluent turbidity dropping rapidly, from 38 to 2 NTU in ~10 min. No visible bed expansion yet.
22	1.5	71		12.8	2	5.5		Quantity of fines washing from bed has increased slightly.
31	2.0	70	7.45	14	2.5	2.8/1.8	120	Transient channeling in bed rather than true fluidization. Fines continuing to be washed off.
50	2.5	66		14.7	4.5	1.5	112	More fines come off immediately after flow increase, but water above bed clears up quickly.
59	3.0	64.5	6.89	15.3	3	1.4	88	Incremental flow increase generates few new fines. Degree of bed fluidization is increasing.
83	3.5	63.5		15.8	4.5			Degree of bed fluidization increases rapidly with incremental flow increase. Significant new fines generated.
99	4.0	62	6.97	16.3	4	0.9	52	Fines continue to leave bed. Top of bed is very fluid.
103	5.0	62		19	8	1.7	44	Significant discrete carbon bed particles are exiting with fines.
111	6.0	61.5	6.95	~22	11	1.5		Effective bed washing flow rate has been exceeded. Large numbers of bed particles are exiting with flow.
≥120	2.5	62		15	2.75	0.8		At design flow rate, bed appears well washed. Water above bed is very clear.

Table 3: Fines Flushing Data for Cameron 8x20 Carbon

Elapsed Time [min]	Flow Rate [gpm]	Water Temp [°F]	pH	Bed Depth [in]	Column dP [psi]	Turbidity [NTU]	Effluent Solids [ppm]	Comments/Observations
0	0	69		12.5	0			Carbon has “mushy” appearance. Interstitial water is very cloudy. Many fines fell through the lower bed retention screen.
6	1.0	73	8.5	13.4	0.5	>50	~2300	Many fine air bubbles coming off bed; not seen with other carbons. Water above bed very dark with fines.
13	1.5	72	6.37	14	2	13		Water above bed still very cloudy, getting better.
20	2.0	69.5	7.1	15	2	7	160	A fresh “puff” of fines emerges from the bed with each incremental flow increase.
26	2.5	68.5	7.18	15.8	3.2	4.8	132	More apparent fines at this flow rate than for the other carbons.
31	3.0	67	7.17	~17.5	3.5	5.3	112	Top of bed is becoming very ill-defined; this did not occur with other carbons until much higher flows.
36	3.5	65	7.13	~19	4.2	4.2		Top of bed difficult to discern.
41	4.0	64	7.16	~20	6.0	3.1	64	As above. Large number of discrete bed particles in effluent sample, but they immediately sink to the bottom of the sample container.
46	4.5	63	7.06	~22	6.5	3.0		As above.
51	5.0	62	7.11	~24	8	3.0	60	As above. Efficient bed washing flow rate has been exceeded, based on the large number of carbon particles entrained in the column effluent.
>60	2.5	62		15.8	2	3.2		Upon returning to the design flow rate, the bed has settled down, but the water above the bed is still visibly cloudy. The bed interface (top) remains less clearly defined than for other carbons at design flow.

Table 4: Summary of Fines Washout Data from All Three Carbons

Flow		Calgon Centaur 12x40		Calgon Centaur 8x30		Cameron 8x20	
[gpm]	[gpm/ft ²]	mg/L	% of Total	mg/L	% of Total	mg/L	% of Total
1.0	5.1	300	33.8	188	31.1	2300	81.3
2.0	10.2	112	12.6	120	19.9	160	5.7
2.5	12.7	164	18.5	112	18.5	132	4.7
3.0	15.3	120	13.5	88	14.6	112	4.0
4.0	20.4	92	10.4	52	8.6	64	2.3
4.5	22.9	100	11.2				
5.0	25.5			44	7.3	60	2.0

Figure 2: Fines Washout Plot for Calgon Centaur 12x40 Carbon

Figure 3: Fines Washout Plot for Calgon Centaur 8x30 Carbon

Figure 4: Fines Washout Plot for Cameron 8x20 Carbon

4.2 TEST 2: Bed Expansion During Upflow

The test procedure is described in detail in Appendix A. The test column was loaded with a 12-inch depth bed of pre-soaked carbon. Beginning at zero flow, upflow through the column was increased incrementally to a maximum of 5 gpm. Bed expansion was measured at each flow increment. Table 5 shows the raw data taken during this testing.

Table 5: Carbon Bed Expansion Data					
Column Flow Rate [gpm]	Superficial Velocity [gpm/ft ²]	Bed Depth [in] and % Expansion			
		Centaur 12x40 (T _{Avg} = 63.5°F)	Centaur 8x30 Run #1 (T _{Avg} = 67.9°F)	Centaur 8x30 Run #2 (T _{Avg} = 64.1°F)	Cameron 8x20 (T _{Avg} = 66.5°F)
0	0	13.5 (0%)	13.0 (0%)	13.3 (0%)	13.0 (0%)
1.0	5.1	14.0 (3.7%)	13.2 (1.5%)	13.5 (1.9%)	13.4 (3.1%)
1.5	7.6	14.6 (8.1%)	13.5 (3.8%)	13.8 (3.8%)	14.0 (7.7%)
2.0	10.2	15.4 (14.1%)	14.0 (7.7%)	14.1 (6.4%)	14.8 (13.8%)
2.5	12.7	17.5 (29.6%)	14.7 (13.1%)	14.7 (10.9%)	15.5 (19.2%)
3.0	15.3	19.5 (44.4%)	15.5 (19.2%)	15.0 (13.2%)	16.5 (26.9%)
3.5	17.8	21.3 (57.4%)	15.8 (21.5%)	15.5 (17.0%)	18.0 (38.5%)
4.0	20.4	23.5 (74.1%)	16.2 (24.6%)	16.3 (23.0%)	19.5 (50.0%)
4.5	22.9	26.5 (96.3%)	17.0 (30.8%)	17.5 (32.1%)	21.0 (61.5%)
5.0	25.5	30.0 (122.2%)	18.5 (42.3%)	18.5 (39.6%)	23.5 (80.8%)

Two runs were performed for Calgon Centaur 8x30 carbon. Run #1 was conducted with increasing incremental flow rate as for the other two carbons, while the data for Run #2 was taken immediately after Run #1 as the column flow was incrementally decreased. The decision to do so was made on the spur of the moment, with no specific intent other than to see how the two data sets would compare. Note that at the end of Run #2, the bed did not settle all the way down to the starting depth of Run #1. The percent expansion values for Run #2 are based on the Run #2 ending bed depth, rather than the Run #1 starting bed depth. Nevertheless, the two sets of data agree well, indicating that there is little, if any, hysteresis in the carbon bed behavior.

The percent bed expansion data is plotted in Figures 5 through 8.

Note that the Calgon Centaur 8x30 plots both exhibit a slight “hump” at superficial velocities of 15.3 gpm/ft² (Run #1, Figure 6) and 12.7 gpm/ft² (Run #2, Figure 7). No explanation is readily

apparent, although it can be theorized that for this particular carbon product, the bed undergoes some type of flow regime transition, perhaps as the degree of bed fluidization increases.

As expected, Calgon Centaur 12x40 exhibited the greatest degree of bed expansion for any given flow rate. However, the surprise is that Cameron 8x20 expanded more than Calgon 8x30. Given that the densities of the two carbons are roughly equal, the 8x20 product's coarser mesh range should have produced somewhat less bed expansion than the 8x30 product. However, it was observed earlier in the test that Cameron 8x20 appeared to contain a wider range of mesh size carbon particles than either of the Calgon products. Hence, it is likely that this disparity between the two brands of carbon, despite their relative mesh range ratings, is responsible for the unexpected behavior of the Cameron carbon.

Figure 5: Bed Expansion Plot for Calgon Centaur 12x40 Carbon

Figure 6: Bed Expansion Plot #1 for Calgon Centaur 8x30 Carbon

Figure 7: Bed Expansion Plot #2 for Calgon Centaur 8x30 Carbon

Figure 8: Bed Expansion Plot for Cameron 8x20 Carbon

4.3 TEST 3: Peroxide Destruction Kinetics

The test procedure is described in detail in Appendix A. The three carbons were tested on a laboratory scale to determine their relative rates for peroxide destruction. In all tests, the water temperature was approximately 20 °C and the pH was approximately 5.0.

The tests were carried out in the 306E Building to eliminate possible interference from mercury lamps in the 305 Building. Mercury lights of the type used in the 305 Building radiate an energy line that catalyzes hydrogen peroxide decomposition by photolysis.

Hydrogen peroxide solutions for this test were made from 3% peroxide solution diluted to the desired concentration with 18 megohm/cm water obtained in the 305 Building. Peroxide concentration determinations were performed with colorimetric test kits produced by CHEMetrics, Inc., which use a thiocyanate reagent to produce the observed color. The CHEMetrics test ampules are precalibrated and are read by comparison with a supplied set of color standards. Since the color is determined by the human eye, tests were read by two investigators separately and the results compared. The investigators had a high degree of agreement on determining the concentration by the color comparator method.

Table 6 shows the raw kinetics data for each carbon tested. Two of the tests had initial peroxide concentrations of 300 mg/L, while the third test began with 200 mg/L. Since the purpose of the testing was to determine kinetics data, and since samples were taken frequently (every 2 minutes

between vigorous agitation), it was believed that the difference in initial concentrations would not diminish the usefulness of the test data.

Table 6: Peroxide Destruction Kinetic Data			
Time [min]	Peroxide Concentration [ppm]		
	Calgon Centaur 12x40	Calgon Centaur 8x30	Cameron 8x20
0	300	200	300
2	75	150	130
4	40	100	125
6	15	60	100
8	6	30	60
10	2	22	40
12	0.8	15	30
14	0.2	9	15
16		5	10
18		2	9
20		0.9	7
22		0.4	3.5
24			2.5

Figures 9 through 13 show non-logarithmic and semi-logarithmic plots of the kinetic data. Data for the Calgon Centaur carbons give good indications of a non-stoichiometric (i.e., catalytic) reaction. The Cameron carbon, on the other hand, shows a response more typical of a stoichiometric reaction in which reaction sites on the carbon are irreversibly consumed, rather than remaining unaffected as in a true catalytic reaction. Although new sites may be exposed through breakup and attrition of the carbon particles, the peroxide destruction reaction ultimately shows a permanent decrease as the carbon itself is consumed by peroxide.

From this kinetic data, an attempt was made to determine the order of the peroxide destruction reaction for each carbon. While the semi-logarithmic plots for the Calgon Centaur carbons closely approximate straight lines, the Cameron carbon does not, which indicates something other than a first order reaction. A linear regression was carried out for the data from each carbon, the results of which are presented in Table 7. It is possible that the Cameron carbon is exhibiting a combination of catalytic and stoichiometric reactions.

Figure 9: Peroxide Destruction Kinetics Data for Calgon Centaur 12x40 Carbon (Non-logarithmic Plot)

Figure 10: Peroxide Destruction Kinetic Data for Calgon Centaur 12x40 Carbon (Semi-logarithmic Plot)

Figure 11: Peroxide Destruction Kinetics Data for Calgon Centaur 8x30 Carbon (Non-logarithmic Plot)

Figure 12: Peroxide Destruction Data for Calgon Centaur 8x30 Carbon (Semi-logarithmic Plot)

**Figure 13: Peroxide Destruction Kinetics Data for Cameron 8x20 Carbon
(Non-logarithmic Plot)**

**Figure 14: Peroxide Destruction Kinetics Data for Cameron 8x20 Carbon
(Semi-logarithmic Plot)**

Table 7: Linear Regression Data and Calculated Reaction Rate for Kinetics			
Carbon	Linear Regression Equation for $\{\ln C/C_o\}$	r^2	Rate (min^{-1})
Calgon Centaur 12x40	$-0.50T + 0.841$	0.99	0.50
Calgon Centaur 8x30	$-0.28T + 0.38$	0.98	0.28
Cameron 8x20	$-0.19T - 0.11$	0.98	0.19

This data indicates that Calgon Centaur 12x40 has nearly double the peroxide destruction efficiency of Calgon Centaur 8x30, and over 2.5 times the efficiency of Cameron 8x20. It was anticipated that Calgon Centaur 12x40 would outperform the other products because of its greater available surface area per unit volume and mass, but the relative degree of its superiority was greater than expected.

Based on this data, if reaction kinetics is the primary consideration, Calgon Centaur 12x40 should be the carbon of choice for the ETF peroxide decomposers.

4.4 TEST 4: Pilot Scale Peroxide Destruction Efficiency

The test procedure is described in detail in Appendix A. Approximately 20 gallons of peroxide solution was pumped through a bed of each carbon in the test column at 2.5 gpm to simulate the design superficial velocity. The inlet peroxide concentration was adjusted as closely as possible to 200 ppm. The temperature averaged 22 °C, while the bulk solution pH was adjusted to between 5.2 and 5.8 with dilute sulfuric acid.

Samples of the column influent (Sample Port 1) and effluent (Sample Port 2) were taken roughly every two minutes. Approximately one column hydraulic retention time was allowed between each influent and effluent sample set.

Table 8 shows the raw data for this testing. This data includes a “blank” run, with no carbon in the column, which was performed to ensure that there was no background peroxide destruction caused by any equipment on the test skid. The carbon bed support screen and flow distributor plate at the bottom of the column were made of stainless steel, and there was some concern that this alloy could have a slight catalytic effect for peroxide destruction. However, no such effect was observed.

In Table 9, the percentage of peroxide destruction across the test column has been calculated for each pair of influent/effluent samples, and an overall average destruction percentage is given for each carbon and the blank.

**Table 8: Raw Data for Pilot Scale Peroxide Destruction Testing
(ND = Not Done)**

Sample No.	Peroxide Concentration [ppm]							
	Calgon Centaur 12x40		Calgon Centaur 8x30		Cameron 8x20		Blank	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
1	200	60	175	75	200	75	180	180
2	200	50	200	60	200	90	180	180
3	200	40	200	40	210	100	180	180
4	200	60	200	30	220	110	180	180
5	200	55	210	40	ND	ND	180	180
6	200	55	210	40	ND	ND	180	180

**Table 9: Percent Peroxide Destruction Across Test Column
(ND = Not Done)**

Sample	Calgon Centaur 12x40	Calgon Centaur 8x30	Cameron 8x20	Blank
1	70	57	63	0
2	75	70	55	0
3	80	80	52	0
4	70	85	50	0
5	73	81	ND	ND
6	73	81	ND	ND
Averages:	73.5	75.7	55.0	0

Calgon Centaur 8x30 was the best performer in this test, appearing slightly better than the same brand of 12x40 carbon. This is surprising in light of the Test 3 results, where Centaur 12x40 showed a peroxide destruction reaction rate nearly twice that of Centaur 8x30. However, the difference of only 2.2 percentage points between the two carbons should probably be taken as negligible, considering the margin of error inherent in the colorimetric peroxide concentration analysis. Further, it is possible that the bed depth of both the Centaur 12x40 and 8x30 were in excess of that required to achieve good peroxide destruction. Other factors, such as the bed flow regime or limited residence time, may also have equalized the apparent performance of these two carbons.

Most noteworthy during this test was the degree of gas bubble formation within the carbon bed as the peroxide underwent destruction. These oxygen bubbles tended to attach themselves to carbon particles, thus providing a buoyancy which lofted many particles up to the column effluent where they were entrained and lost from the bed. All three beds of carbon had been previously washed in Test 1, and were further washed during the bed expansion of Test 2. Hence, this lofting and loss of carbon during the peroxide destruction test cannot be attributed to inadequate fines removal.

The lofting effect was most pronounced for Cameron 8x20 carbon, a bit better for Calgon Centaur 12x40 carbon, and least for Calgon Centaur 8x30 carbon. However, all three carbons were seriously affect by this mechanism; even the Centaur 8x30 exhibited the potential for significant bed loss over time.

The particle lofting due to gas bubble formation and attachment appears to be inherent in the peroxide destruction process with activated carbon. Mechanical methods will need to be employed to minimize carbon loss from the ETF peroxide decomposer columns.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The following recommendations are based on the results of this testing. They are made with the realization that although laboratory and pilot scale data can indicate definite trends, the effect of scaling up test results to process scale equipment and conditions introduces significant uncertainty.

Fines removal from new carbon in the ETF peroxide decomposers should be performed at greater than the design process flowrate of 85 gpm. A full-scale column flow rate of 120 to 140 gpm should provide adequate flushing velocity for fines removal without excessive carbon loss from the bed. The pilot scale column appeared to be well flushed after about one hour, but the sheer volume of carbon contained in the full size columns may require a proportionally longer flush time. A initial flush time of four to six hours would be advisable, followed by sampling of the column effluent to observe the amount of fines that continue to be removed. If a finer mesh of carbon, such as Calgon Centaur 12x40, is in use, it is advisable to stay below 150 gpm per column in order to avoid excessive bed expansion and loss of carbon.

Visual observations, as well as the kinetic data, indicate that the bituminous coal based Calgon Centaur is superior in both quality and peroxide destruction efficiency to the coconut shell based Cameron carbon. The Centaur 12x40 has a significant advantage over the Centaur 8x30 for peroxide destruction efficiency under laboratory conditions, but this advantage may narrow in the full scale application as it did in the pilot scale test. Nevertheless, assuming that an adequate mechanical system is in place to counteract the effects of carbon particle buoyancy, this testing indicates that Calgon Centaur 12x40 would be the best choice for use in the ETF decomposer columns.

Depending on how the peroxide decomposers perform during the next ETF campaign, it might be advisable to perform further studies in two areas:

- Investigate the existence and effectiveness of metallic or ceramic catalysts for peroxide destruction. Such catalysts, if they exist, would have the advantage of negligible fines production and potentially long life due to their material characteristics. Extended catalyst life will reduce the amount of solid waste generated by operation of the columns. Instead of annual carbon changes, it might be possible to have a catalyst that would last five to ten years, or longer, and would be sufficiently robust to withstand vigorous chemical cleaning. Also, the greater density of a metal or ceramic catalyst would likely eliminate the gas bubble buoyancy effect.
- Fine-tune the column discharge header design to reduce the quantity of carbon carryover due to gas bubble buoyancy. This could be done via pilot scale studies of discharge header behavior.

Both of the above items could be done with existing test equipment. A considerable amount of craftsmanship was devoted to the design and construction of the pilot scale column test skid, and this equipment remains in storage at the 305 Building. Any further testing would not need to fund the fabrication of new test equipment, thus, all funding could go toward the performance of actual testing and the analysis of data.

APPENDIX A

CARBON EVALUATION TEST EQUIPMENT AND PROCEDURE

APPENDIX A

CARBON EVALUATION TEST EQUIPMENT AND PROCEDURE

1.0 EQUIPMENT

Column - Carbon will be tested in a vertical, 6-inch diameter clear plastic column with a minimum height of five column diameters above the bottom of the carbon bed. The column inlet flow will be distributed/diffused ahead of the carbon bed to obtain a uniform velocity profile through the bed.

Carbon Bed - The carbon bed depth will be two column diameters (12 inches) packed (i.e., as loaded, not expanded by upflow).

Pump and Flowmeter - A pump with a variable output range of 0 to approximately 10 gpm will provide the fluid motive force for peroxide destruction testing. Flow will be measured with a rotameter calibrated in increments of 0.5 gpm or less.

Peroxide Solution Feed and Receiving Tanks - Tankage with a minimum useable volume of 20 gal each will be used to support the pilot scale peroxide destruction testing.

Water Source - A deionized (DI) water source will be needed to prepare peroxide solutions. A connection to a tap (sanitary) water source will be needed for the fines washing and bed expansion tests. All tests will be conducted with water at ambient temperature.

Sample Ports - Sample ports will be provided at the column inlet and outlet for various sampling and monitoring purposes. The quantity of carbon fines column outlet stream during carbon fines washing will be measured using gravimetric methods, and possibly with a bench turbidity instrument. This monitoring will help determine the optimum fines washing conditions. Also, both the column inlet and outlet sample ports will be used during pilot scale peroxide testing to measure the degree of peroxide destruction across the column.

In-Line Filter - The column discharge line will contain a 25- μ m nominal filter to collect larger carbon particles that wash off the bed. The filter housing will be transparent to allow observation of the element, which will be changed for each carbon. The filter will have upstream and downstream isolation valves to facilitate filter element changes, and there will be a valved filter bypass that can be opened if the filter dP becomes excessive or when flow through the filter is not needed.

Thermocouple and Readout - A temperature element will be used to monitor column inlet water temperature.

Measuring Stick - A yardstick or meter stick will be used to measure carbon bed depth and expansion.

Stopwatch - A stopwatch will be used to track and record the elapsed time during test activities.

Peroxide Test Kits - CHEMetrics colorimetric peroxide test kits K-5510 (range 0-10 ppm), K-5510D (range 0-250 ppm), and K-5510B (range 0-1000 ppm) will be used to monitor peroxide concentration during kinetics, isotherm, and pilot scale peroxide destruction testing.

pH Probe and Meter - Column outlet pH will be periodically monitored by measuring the pH of samples taken from the column outlet sample port.

Note: The kinetics and isotherm testing requires additional laboratory equipment. See that section for specific equipment needs.

1.1 Equipment Configuration (Test Skid)

Figure A-1 shows the schematic for the test skid that will be used for Test 1 (fines removal), Test 2 (bed expansion), and Test 4 (pilot scale peroxide destruction). These tests are described below.

2.0 CARBON PREPARATION PRIOR TO TESTING

New carbon will be soaked in ambient temperature tap water for a minimum of 72 hours to ensure ample time for proper degassing and wetting.

All carbon beds should be as close to the same depth (two column diameters, or 12 inches) as possible.

3.0 TEST 1: Time Required to Wash Fines from New Carbon

Tap water will be run through the column at varying rates to determine the fluid velocity and time required to remove fines from a bed of new carbon.

Refer to the schematic in Figure A-1. The valve lineup is as follows:

Test Skid Valve Lineup for Test 1	
Valve	Position
Peroxide feed tank pump discharge	Closed
Sanitary water inlet	Open
Filter inlet	Open
Filter outlet	Open
Filter bypass	Closed
Peroxide receiving tank inlet	Closed
Discharge to drain	Open

- (a) Connect a tap water source to the sanitary water inlet hose connection (See Fig. 1). Route the skid drain to an approved building drain receptacle.
- (b) Start with a 12-inch depth bed of wetted (presoaked) carbon loaded into the test column. Record the actual carbon bed depth.
- (c) Connect the column inlet to the tap water source, with the rotameter in line to monitor the flow rate. If necessary, install a new element in the column discharge line filter, and open the filter inlet and outlet valves. Close the filter bypass valve so that all flow is directed through the filter. Ensure that a new filter element is installed at the beginning of this test.
- (d) Monitor and record water temperature, differential pressure, and column discharge pH during this test.
- (e) Start the stopwatch and bring the column flow up to 1 gpm. Thereafter, increase flow in 0.5-gpm increments. (See Table 1 for flow rate vs. superficial velocity for a 6-inch diameter column.) Allow the column flow dynamics to stabilize at each incremental flow rate. Record each flow rate, and any observations. Obtain a sample from the column outlet sample port at each incremental flow rate, and perform pH measurement and gravimetric analysis (total solids) on the sample. Also perform turbidity measurements on the samples, if turbidity instrumentation is available.
- (f) Stop increasing the flow rate through the column when significant quantities of larger carbon particles become entrained in the column outlet stream. Qualitatively note the quantity of coarse carbon particles that have been trapped in the 25- μ m filter.

- (g) Stop the water flow and stopwatch. Allow the carbon bed to settle completely. Record the elapsed time and the final bed depth.
- (h) Proceed to Test 2 with the carbon already in the column. The carbon may remain in the column for an extended period between tests, but the column water level must remain above the bed so that the carbon is continuously wetted.

4.0 TEST 2: Bed Expansion During Upflow

Tap water will be run through the column at varying flow rates to measure the bed expansion of washed carbon.

The valve lineup and skid connections to a tap water source and a drain are the same as for Test 1.

- (a) Record the carbon bed depth with zero flow through the column (should be the same as at the end of Test 1).
- (b) Beginning 1 gpm, increase flow in 0.5-gpm increments. (See Table 1 for flow rate vs. superficial velocity for a 6-inch diameter column.) Allow the column flow dynamics to stabilize at each incremental flow rate. Record the flow rate, the bed depth in inches, and any observations. Also, periodically record the column inlet water temperature.
- (c) Stop increasing the flow rate when discrete carbon bed particles are observed lofting out of the column. Record this flow rate, and any observations.
- (d) Reduce flow to zero. Record the final bed depth.
- (e) Valve out the filter. Remove the element, bag it in clear plastic, and label it with the appropriate carbon brand and mesh size for future reference.
- (f) Test 4 will be performed after Test 3 below. Test 3 entails laboratory work and will not use the test skid. The carbon may remain in the column for an extended period, but the column water level must remain above the bed so that the carbon is continuously wetted.

5.0 TEST 3: Peroxide Destruction Kinetics

Isotherm and reaction rate testing will be conducted on all carbons.

5.1 General

- (a) Obtain about 100 mL of each carbon (washed and wetted) from the material used in Tests 1 and 2. The carbon will be kept in closed containers, and covered with water so that it remains wetted.

5.2 Kinetics Testing

- (a) The reaction will be carried out as a batch reaction. Prepare the carbon to be tested as outlined above in section 1.
- (b) Weigh out 50 grams of carbon and add to 100 mL of DI water as in 1B. Add enough peroxide to give a final concentration of 200 mg/L.
- (c) Beginning at time $t=0$, begin sampling residual peroxide every minute for 10 minutes or until a less than value is observed.
- (d) Tabulate the data in the following format:

Time, min	[A] mol/L	$-\log[A_t/A_0]$	$1/[A]$

- (e) Plot $-\log[A_t/A_0]$ versus t to determine if the reaction is first order. If the plot does not result in a straight line, then plot $1/[A]$ versus t to determine if the reaction is a second order.

6.0 TEST 4: Pilot Scale Peroxide Destruction Efficiency

The purpose of this test is to confirm the kinetics and isotherm data from Test 3 above, using the bench-scale column for relative peroxide destruction performance testing of the three carbons.

Refer to the schematic in Figure A-1. The valve lineup is as follows:

Test Skid Valve Lineup for Test 4	
Valve	Position
Peroxide feed tank pump discharge	Open
Sanitary water inlet	Closed
Filter inlet	Closed
Filter outlet	Closed
Filter bypass	Open
Peroxide receiving tank inlet	Open
Discharge to drain	Closed

DI water will be used to make up the peroxide solutions for this test, in order to simulate the ultra-pure water conditions that will exist when the ultraviolet oxidation system is located downstream of the reverse osmosis system during groundwater processing.

- (a) Make up approximately 20-gal of DI water containing 200 ppm peroxide adjusted to a pH of 5 to 6 with dilute H_2SO_4 . Measure and record the actual peroxide concentration with the appropriate range CHEMetrics peroxide test kit, which should be K-5510D (range 0-250 ppm).
- (b) Simultaneously start the stopwatch and turn on the peroxide solution feed pump. Immediately adjust the column flow rate to 2.5 gpm, and periodically monitor and record the column influent and effluent peroxide concentration by sampling from the column inlet and outlet sample ports (Sample Ports 1 and 2, respectively, in Figure A-1).
- (c) Continue pumping until the peroxide solution feed pump begins to cavitate, indicating that the feed tank has run dry. Stop the stopwatch and record the elapsed time.
- (d) Record any pertinent observations made during this test.

7.0 CONTROL TEST FOR SYSTEM PEROXIDE DESTRUCTION

Follow the procedure outlined in Sec. 6.0 (Test 4), except that no carbon is to be used. This is a “blank” test to determine if the system configuration or materials have a notable effect on peroxide destruction rate, and to establish any “background” rate of peroxide destruction that may need to be subtracted from the peroxide destruction performance of the carbon.

After performing this test, evaluate whether a significant background peroxide destruction rate exists.

8.0 CALCULATIONS

8.1 ETF Decomposer Column Calculations

Cross-sectional area of ETF decomposer column (3 ft diameter):

$$A_{\text{ETF}} = (\pi/4)(3 \text{ ft})^2 = 7.07 \text{ ft}^2$$

Superficial velocity in ETF decomposer column @ 85 gpm (max process flow):

$$\begin{aligned} S.V. &= (85 \text{ gal/min})/(7.07 \text{ ft}^2) = 12.0 \text{ gpm/ft}^2 \\ &= (12.0 \text{ gpm/ft}^2)(1 \text{ ft}^3/7.48 \text{ gal})(1 \text{ min}/60 \text{ s}) = 0.027 \text{ ft/s} \end{aligned}$$

Packed bed fluid residence time in ETF decomposer column @ 85 gpm and 6 ft bed depth:

$$t_R = [(6 \text{ ft})/(0.027 \text{ ft/s})](1 \text{ min}/60 \text{ s}) = 3.70 \text{ min}$$

8.2 Test Column Calculations

Cross sectional area of 6-in test column:

$$A_{\text{test}} = (\pi/4)(0.5 \text{ ft})^2 = 0.196 \text{ ft}^2$$

Test column flow rate necessary to obtain superficial velocity of 12 gpm/ft²:

$$\text{Flow} = (12.0 \text{ gpm/ft}^2)(0.196 \text{ ft}^2) = 2.36 \text{ gpm} \approx 2.4 \text{ gpm}$$

Packed bed fluid residence time in test column @ 2.4 gpm:

$$t_R = (1 \text{ ft})(0.196 \text{ ft}^2)(7.48 \text{ gal/ft}^3)/(2.4 \text{ gal/min}) = 0.61 \text{ min} = 37 \text{ sec}$$

TABLE 1: Flow Rate vs. Superficial Velocity for 6-inch Column		
Flow Rate [gpm]	Superficial Velocity [gpm/ft ²]	Superficial Velocity [ft/s]
0.5	2.55	0.006
1.0	5.09	0.011
1.5	7.64	0.017
2.0	10.2	0.023
2.4	12.0	0.027
2.5	12.7	0.028
3.0	15.3	0.034
3.5	17.8	0.040
4.0	20.4	0.045
4.5	22.9	0.051
5.0	25.5	0.057
5.5	28.0	0.062
6.0	30.6	0.068
6.5	33.1	0.074
7.0	35.7	0.079
7.5	38.2	0.085
8.0	40.7	0.091
8.5	43.3	0.096
9.0	45.8	0.102
9.5	48.4	0.108
10.0	50.9	0.113

Figure 1: Test Skid Schematic for Carbon Tests 1, 2, and 4

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