

ANNUAL TECHNICAL PROGRESS REPORT NO. 2

ENHANCED OXIDATION CATALYSTS FOR WATER RECLAMATION

DATE: September 9, 1999

CONTRACT NO: NASW-97016

ISSUING AGENCY: Goddard Space Flight Center
Headquarters Acquisition Branch
Code 210.H
Greenbelt, MD 20771
Robin Morton, Contract Administrator
301/286-4270

CONTRACTOR: Environmental & Life Support Technology, Inc.
6600 E. Lookout Dr.
Parker, CO 80138
Clifford D. Jolly, Principal Investigator
303/840-4221

1.0 OVERVIEW

Based on results from the last reporting period, four treated catalyst materials were selected for column testing to quantify the long-term performance of these materials against one untreated catalyst material and one commercially-available (Engelhard) catalyst material. All column loadings were 3% Pt on Barnebey-Sutcliffe 208-C Granular Activated Carbon to provide a standard test set and a sufficient Pt loading to create a significant challenge to the oxidation-resistance treatments. The challenge solution was 30 mg/l total organic carbon as ethanol.

Three catalyst treatments (based on borate and boron nitride chemistries) exhibited significantly improved resistance to oxidative degradation at 250 F and 60 psig O₂ pressure. Based on these results the borate and boron nitride treatment processes were further investigated by applying them to additional support materials and varying the manufacturing procedure.

2.0 CURRENT STATUS AND WORK PERFORMED TO DATE

Testing was accomplished using a flow-through design representative of a liquid phase catalytic process that uses molecular oxygen as the oxidant. Multiple catalyst beds can be tested at the same flowrate and the same temperature using this test stand, allowing direct and simultaneous comparison of different treatment techniques. Pre-heating the influent stream and utilizing a 12" x 2" ceramic cylinder heater that does not directly contact the stainless steel reactor walls was used. The test apparatus is shown in Figure 1. Figure 2 shows the theoretical (Henry's Law) concentration of dissolved oxygen vs pressure. Test conditions were defined as follows:

- empty bed contact time 5 minutes
- reactor pressure 60 psig O₂
- catalyst volume 10 cc
- temperature 250F

Table 1 shows the materials that were selected for long-term tests (the screening test data were reported previously). These materials are:

| | |
|-------|---|
| U-301 | untreated 3% Pt on Barnebey-Sutcliffe 208-C carbon |
| BB-3A | borate-treated 3% Pt on 208-C |
| BB-P1 | mixed boron nitride/phosphate-treated 3% Pt on 208-C |
| E-235 | commercial material obtained from Engelhard Corporation based on their best material for these process conditions |
| BP-2A | phosphate-treated 3% Pt on 208-C |
| BB-3B | borate-treated 3% Pt on 208-C |

Tests were previously conducted on analogous materials to assess both the materials platinum loss and their catalytic activity. Measurement of catalyst physical stability was attempted by two methods 1) by measuring platinum loss into solution, and 2) by stopping the tests and drying and re-weighing the catalysts at four month intervals. Pt loss to solution was found at the end of the last reporting period to correlate poorly to actual carbon support weight loss. Therefore, the catalyst weight loss was determined during this reporting period by halting the tests, drying the materials at 120 C under UHP nitrogen, removing the catalyst from the stainless steel housing, weighing the catalyst, re-loading the housing and restarting the test. These data are shown in Figure 3.

Table 1. Platinum Loss Screening Test Data

| <u>Catalyst</u> | <u>Platinum Loading (%)</u> | <u>Pt Loss (%)</u> |
|-----------------------------|-----------------------------|--------------------|
| U-301 (untreated control) | 3 | 6.86 |
| BB-3A (oxidation-protected) | 3 | 0.69 |
| BB-P1 (oxidation-protected) | 3 | 0.83 |
| E-235 (oxidation-protected) | 3 | 0.40 |
| BP-2A (oxidation-protected) | 3 | 1.31 |
| BB-3B (oxidation-protected) | 3 | 0.52 |

Table 2. Long-term Dynamic Column Tests

Test Data through May 30,
1999

Test Conditions:

| | |
|---------------|------------------------|
| Temperature | 250F |
| O2 Pressure | 60 psig |
| Flowrate | 2 ml/min |
| Feed Solution | 30 mg/l TOC as Ethanol |
| Column Size | 10 cc |
| Test Duration | 242 days |

| Column No. | Catalyst mass (g) | | | | | |
|----------------------------------|-------------------|--------------|--------------|--------------|--------------|--------------|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| <u>Through</u> <u>put (L)</u> | <u>U-301</u> | <u>BB-3A</u> | <u>BB-P1</u> | <u>E-235</u> | <u>BP-2A</u> | <u>BB-3B</u> |
| Initial | 4.57 | 4.96 | 5.41 | 4.65 | 4.84 | 4.77 |
| 257 | 3.49 | 4.41 | 4.58 | 3.58 | 4.37 | 4.32 |
| 524 | 1.38 | 4.26 | 4.23 | 1.84 | 3.44 | 4.08 |
| 722 | 0.89 | 3.90 | 4.01 | 1.01 | 2.15 | 3.88 |

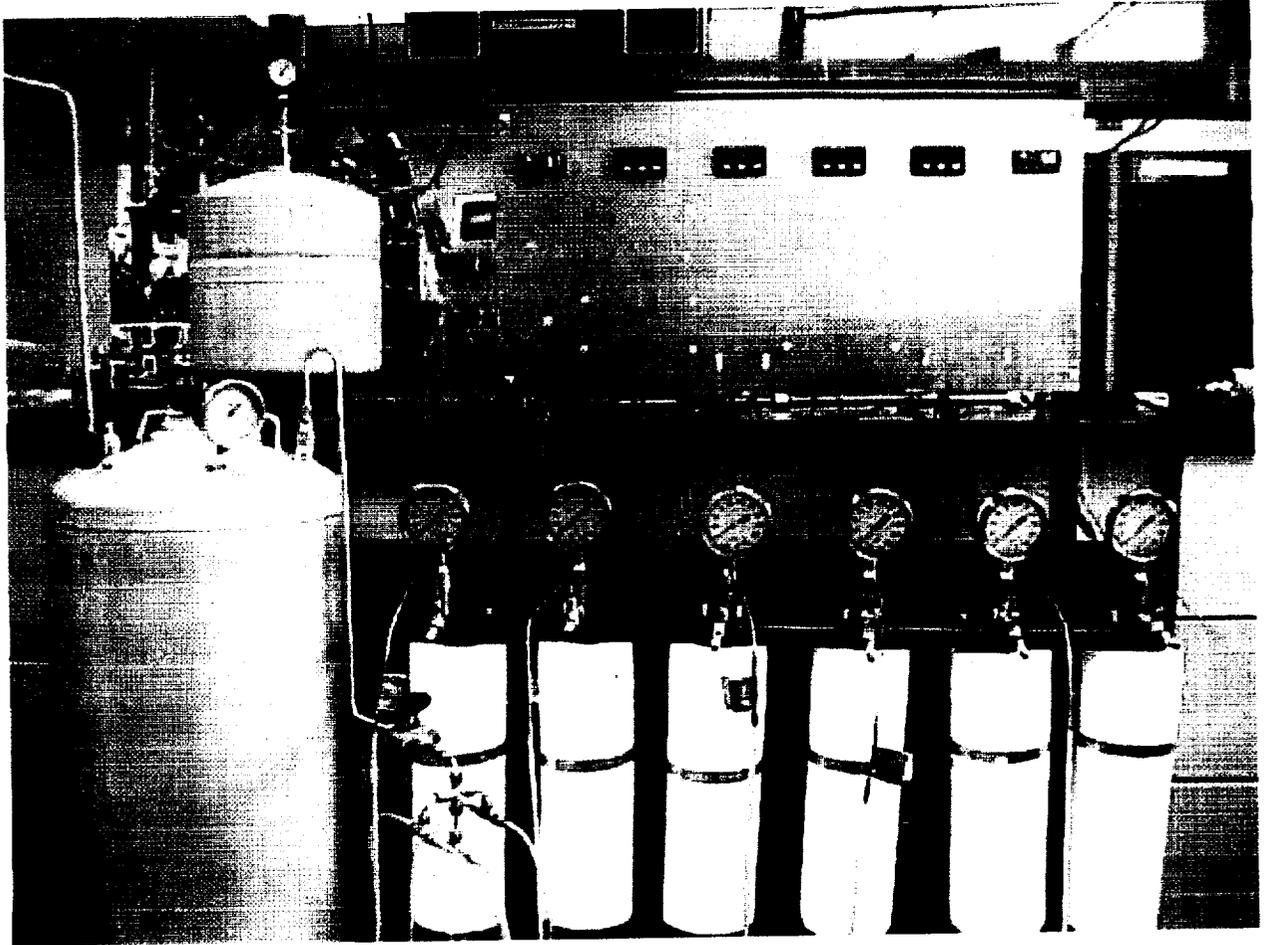


Figure 1. Catalyst Parallel Test Stand

Figure 2. Dissolved Oxygen Concentration vs Pressure by Henry's Law

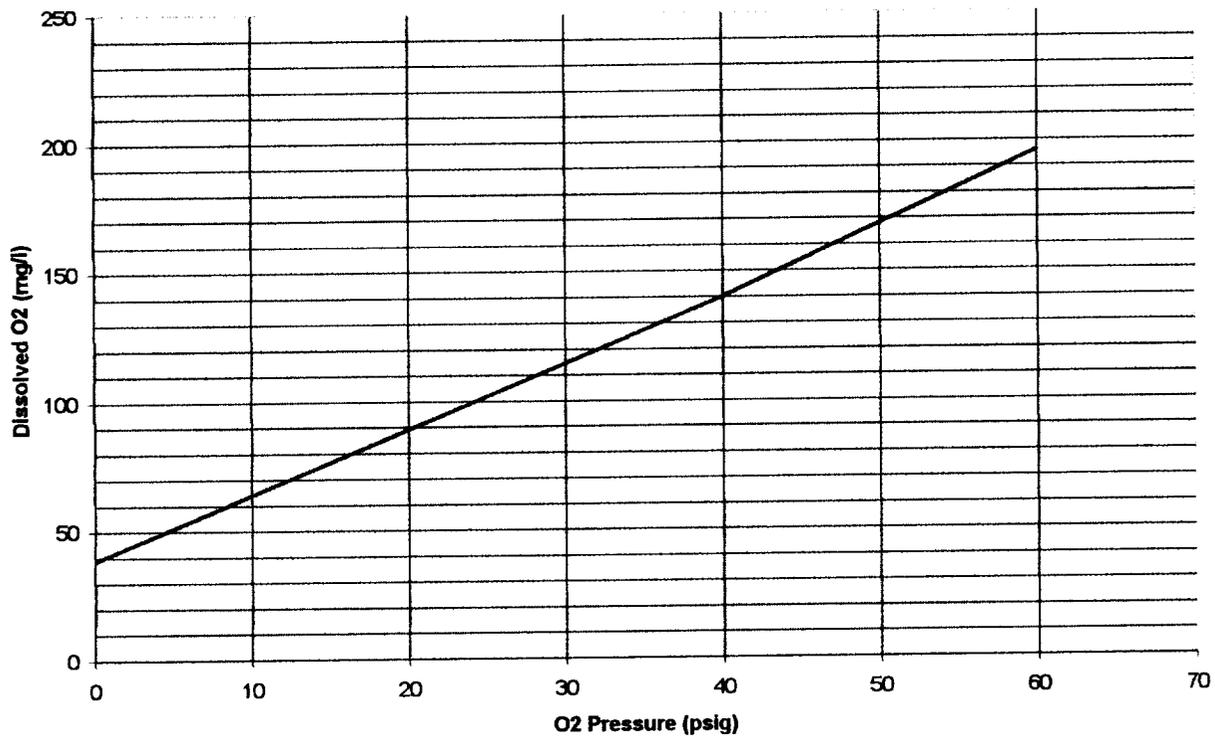


Figure 3. Catalyst Weight Loss vs Throughput

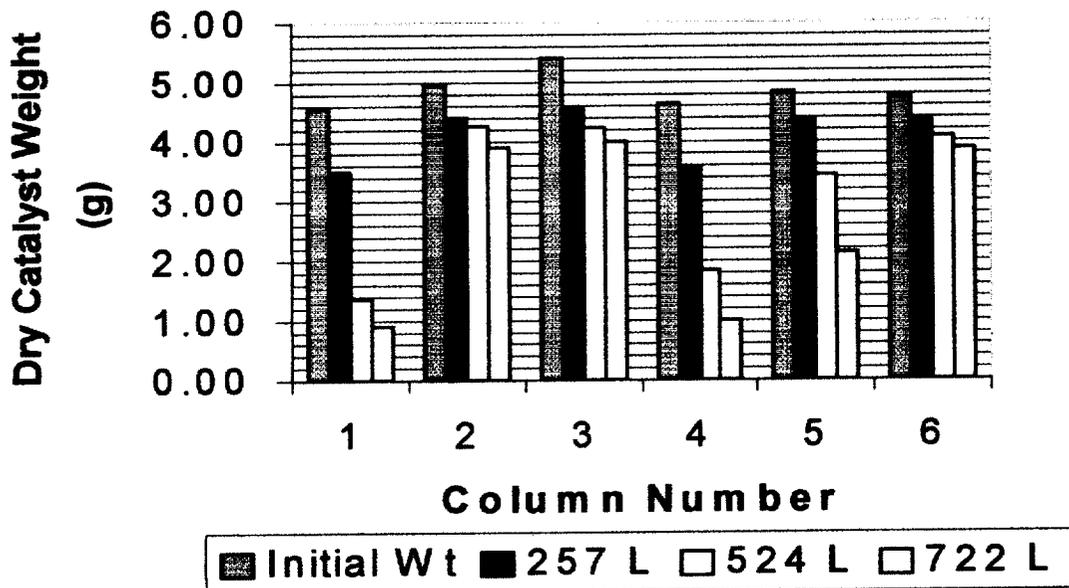
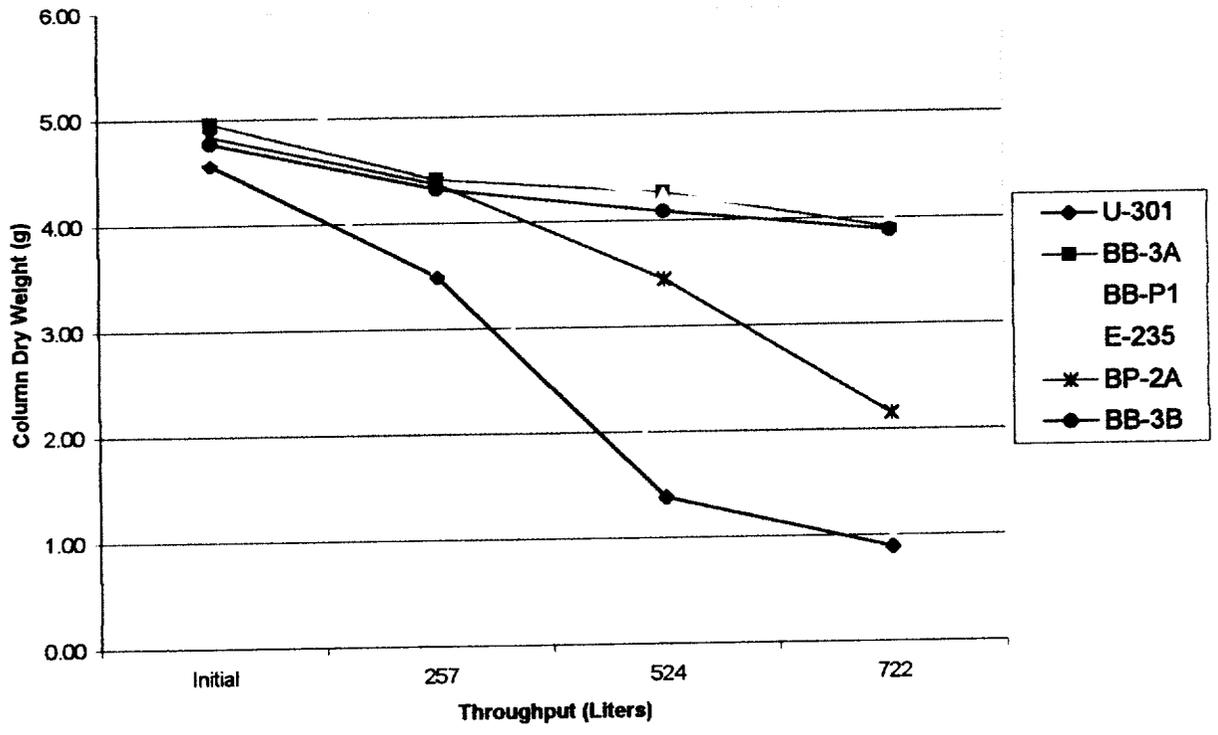


Figure 4. Catalyst Weight Loss vs Throughput



3.0 ANALYSIS OF RESULTS and FUTURE WORK

The test data during the second year of the project has identified three catalyst treatments (based on borate and boron nitride chemistries) that exhibit significantly improved resistance to oxidative degradation at 250 F and 60 psig O₂ pressure. The remaining treated materials (based on phosphate treatment chemistry) exhibited good early performance but began to attain a rate of oxidative degradation more similar to untreated materials during the test period. Based on previous work as well as these test data, it is postulated that the phosphate-based coatings have a significant level of solubility under aqueous-phase reactor conditions that limits their long-term effectiveness. The phosphate-based treatments are more effective for gas-phase operation, but less suitable (although according to these data still better than untreated catalysts) for aqueous-phase contaminant oxidations. Based on these results the borate and boron nitride treatment processes are suggested for further investigation by applying them to additional support materials and varying the manufacturing procedure, and replacing the phosphate-treated column with an additional borate/boron nitride-treated column.

| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
|--|--|--|--|--|
| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503. | | | | |
| 1. AGENCY USE ONLY (Leave Blank) | 2. REPORT DATE 09SEPT99 | 3. REPORT TYPE AND DATES COVERED Annual Technical Progress Report | | |
| 4. TITLE AND SUBTITLE Enhanced Oxidation Catalysts for Water Reclamation | | | 5. FUNDING NUMBERS NASW-97016 | |
| 6. AUTHOR(S) Clifford D. Jolly | | | | |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Environmental and Life Support Technology, Inc. 6600 E. Lookout Drive Parker, CO 80138 | | | 8. PERFORMING ORGANIZATION REPORT NUMBER | |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Goddard Space FLight Center Greenbelt, MD 20771 | | | 10. SPONSORING/MONITORING AGENCY REPORT NUMBER | |
| 11. SUPPLEMENTARY NOTES | | | | |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT | | | 12b. DISTRIBUTION CODE | |
| 13. ABSTRACT (Maximum 200 words) This effort seeks to develop and test high-performance, long operating life, physically stable catalysts for use in spacecraft water reclamation systems. The primary goals are to a) reduce the quantity of expendable water filters used to purify water aboard spacecraft, b) to extend the life of the oxidation catalysts used for eliminating organic contaminants in the water reclamation systems, and c) reduce the weight/volume of the catalytic oxidation systems (e.g. VRA) used. This effort is targeted toward later space station utilization and will consist of developing flight-qualifiable catalysts and long-term ground tests of the catalyst prior to their utilization in flight. Fixed-bed catalytic reactors containing 5% platinum on granular activated carbon have been subjected to long-term dynamic column tests to measure catalyst stability vs throughput. The data generated so far indicate that an order of magnitude improvement can be obtained with the treated catalysts vs the control catalyst, at only a minor loss (approx 10%) in the initial catalytic activity. | | | | |
| 14. SUBJECT TERMS Physically stable catalysts for use in spacecraft water reclamation systems | | | 15. NUMBER OF PAGES 9 | |
| | | | 16. PRICE CODE | |
| 17. SECURITY CLASSIFICATION OF REPORT unclassified | 18. SECURITY CLASSIFICATION OF THIS PAGE unclassified | 19. SECURITY CLASSIFICATION OF ABSTRACT unclassified | 20. LIMITATION OF ABSTRACT | |