# SORBENT TESTING FOR SOLIDIFICATION OF ORGANIC PLUTONIUM/URANIUM EXTRACTION WASTE

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## ABSTRACT

The US Department of Energy is evaluating various sorbents to solidify and immobilize radioactive and hazardous constituents of the organic fraction of plutonium/uranium extraction (PUREX) process waste at Savannah River Site (SRS). The purpose of the solidification is to provide a cost effective alternative to incineration of the waste. Incineration at the Consolidated Incinerator Facility (CIF) at SRS is currently identified as the treatment technology for PUREX waste. However, the CIF is not in operation at this time, so SRS is interested in pursuing alternatives to incineration for treatment of this waste.

The DOE Western Environmental Technology Office (WETO) in Butte, MT was designated as the facility for conducting the sorbent testing and evaluation for an organic PUREX waste surrogate. MSE Technology Applications, Inc. tested and evaluated two clay and two polymer sorbents with the capability of solidifying organic PUREX waste. A surrogate organic PUREX waste recipe was utilized, and sorbents were tested and evaluated at bench-scale, 5-gallon scale, and 55-gallon scale.

This paper presents experimental results evaluating four sorbent materials including: Imbiber Beads™ IMB230301-R, Nochar A610 Petrobond™, Petroset II™, and Petroset II Granular™. Previous work at SRS indicated that these products could solidify organic PUREX waste on a bench-scale. The sorbents were evaluated using operational criteria, and final wasteform properties. Operational criteria included: sorbent capacity; sorption rate; sorbent handling; and mixing requirements. Final wasteform evaluation properties included: ignitability; thermal stability; and volumetric expansion. Bench-scale tests, 5-gallon tests, and initial 55-gallon tests are complete. This paper summarizes the results of the bench-scale, 5-gallon scale, and 55-gallon scale tests performed to date. Offgas generation/characterization tests and leachability tests are ongoing.

## INTRODUCTION

The U.S. Department of Energy (DOE) is evaluating various sorbents to solidify the organic fraction of plutonium/uranium extraction (PUREX) process waste at the Savannah River Site (SRS). The purpose of the solidification is to provide a cost-effective alternative to incineration of the waste. Incineration at the Consolidated Incinerator Facility (CIF) at SRS is currently identified as the treatment technology for PUREX waste. However, the CIF is not in operation at this time; therefore, SRS is interested in pursuing alternatives to incineration for treatment of this waste (1).

Solidification of the organic fraction of the PUREX waste using sorbents was identified as a promising alternative to incineration. Accordingly, under the direction of DOE's Western Environmental Technology Office (WETO) in Butte, Montana, and SRS, MSE Technology Applications, Inc. (MSE) tested and evaluated four sorbents with the capability of solidifying organic PUREX waste and rendering it nonhazardous for low-level shallow land disposal at SRS or at the Nevada Test Site (NTS). The secondary waste generated from PUREX solidification must comply with Resource Conservation and Recovery Act (RCRA) disposal requirements and the Waste Acceptance Criteria (WAC) for disposal at SRS or NTS.

The PUREX waste is stored in H-Area at the New Solvent Storage Facility at SRS and derives from an organic-aqueous isotope separation process at SRS. The total volume of legacy PUREX waste is 38,000 gallons—25,000 gallons of spent PUREX organic liquid solvent and 13,000 gallons of aqueous waste. Organic PUREX waste, which was the focus of this study, contains n-paraffins and tributyl phosphate in addition to aromatic hydrocarbons and amine compounds (1). A surrogate of organic PUREX waste was used for the testing at MSE.

Initially, four sorbent materials were evaluated: Imbiber Beads IMB230301-R, Nochar A610 Petrobond, Petroset II, and Petroset II-G. Previous work at SRS indicated that the above sorbents were capable of solidifying PUREX waste on a bench scale and recommended further evaluation of the four sorbents (1,2). The sorbents were evaluated using operational criteria, and final wasteform properties. Operational criteria included: sorbent capacity; sorption rate; sorbent handling; and mixing requirements. Final wasteform evaluation properties included: leachability, ignitability; thermal stability; long-term stability; and volumetric expansion. Sorbent evaluation criteria included sorbent handling, sorbent capacity, sorption rate, sorbent composition, dust production potential, mixing requirements, volumetric expansion, and sorbent cost. Process performance criteria included consistency of solidified product, time to solidify, equipment cost, and equipment maintenance.

## OBJECTIVES

The overall goal of this work was to identify a sorbent capable of solidifying PUREX waste that is also compatible with processing equipment and can maintain stability under a variety of conditions that could occur during shipment/storage of the solidified wastes.

Specific objectives of the bench-scale sorbent testing and evaluation were:

- set up a sorbent testing laboratory to provide on-site analysis and direct comparison of sorbents;
- identify recipe(s) for representative surrogates to simulate PUREX wastes;
- identify sorbents that are effective at solidifying surrogate PUREX wastes; and
- determine the sorbent capacity and sorption rate for each sorbent/surrogate combination on a bench scale.

Specific objectives of the 5-gallon and 55-gallon scale sorbent testing and evaluation were:

- verify appropriate waste loading ratios during pretest optimization studies;
- verify optimum waste loading ratios/mixing/addition methods at a 5-gal scale;
- determine volumetric expansion at a 5-gal scale and estimate waste volume generated per 1,000 gal of PUREX treated;
- determine sorption rate for each sorbent/surrogate combination on a 5-gal scale;
- determine ignitability of final wasteforms;
- verify thermal stability of selected wasteforms;
- perform cost evaluation of sorbents based on 5-gal-scale test results to determine cost per 1,000 gal of PUREX waste treated; and
- perform 55-gal scaleup tests with top two performing sorbents based on results of the 5-gal tests.

## MATERIAL DESCRIPTIONS

To perform the testing, a surrogate PUREX recipe and candidate sorbents were identified. The surrogate PUREX recipe was developed at SRS to perform initial sorbent screening tests and should provide a representative comparison with the actual organic PUREX waste requiring solidification. Sorbents to be tested at the MSE test facility in Butte, Montana, were identified with input from SRS personnel, vendors, and literature/internet searches.

### PUREX Surrogate Formulation

The recipe for the PUREX surrogate (Table I) was developed for previously performed sorbent tests at SRS (1,2).

Table I PUREX Surrogate Recipe

| Chemical Name | Weight Percent |
|---|---|
| Tributyl Phosphate | 17.60% |
| Aliphatic Hydrocarbons | |
| Undecane | 8.45% |
| Dodecane | 8.45% |
| Tridecane | 8.45% |
| Tetradecane | 8.45% |
| Aromatic Hydrocarbons | |
| Diethylbenzene | 21.00% |
| Di-isopropylbenzene | 21.00% |
| Aliphatic Amine | |
| Di-n-octylamine | 6.60% |
| Total | 100% |

The surrogate for 55-gal tests was modified to include only kerosene and tributyl phosphate. The cost of the full surrogate is prohibitive at this scale; therefore, small-scale tests were performed to ensure that sorb times were comparable (within 10%). Table II summarizes the surrogate makeup for the 55-gal tests.

Table II PUREX surrogate recipe for 55-gal drum tests

| Chemical Name | Weight Percent |
|---|---|
| Tributyl phosphate | 17.60 |
| Kerosene | 82.40 |
| **Total** | 100.00 |

**Sorbent Descriptions**

Sorbents identified for testing at a bench-scale included:

- Imbiber Beads IMB230301-R, which are composed of spherical alkylstyrene copolymer and are manufactured by Imbibitive Technologies, Inc.;
- Nochar A610 Petrobond, a sorbent composed of proprietary polymer crystals that is manufactured by Nochar, Inc.;
- Petroset II, a modified clay powder stabilizing agent that is manufactured by Fluid Tech, Inc.; and
- Petroset II-G, a modified clay granular stabilizing agent that is manufactured by Fluid Tech, Inc. and does not require mixing during the waste solidification process.

Based on the results of the bench-scale evaluation, Petroset II was eliminated from further consideration. Sorbents identified for testing during the 5-gallon scale tests included: Imbiber Beads; Nochar A610 Petrobond; Petroset II-G. These three sorbents were tested to verify results from the bench-scale study and determine the optimum deployment strategy for each material.

Based on the results of the 5-gallon tests, Imbiber Beads were eliminated from further consideration due to the non-cohesive nature of the final wasteform. Nochar and Petroset II-G were tested at the 55-gallon scale. Table III summarizes the sorbent properties and estimated bulk price for each material.

Table III Sorbent Description, Density, and Estimated Bulk Price

| Sorbent Name | Sorbent Description | Bulk Density ($g/cm^3$) | Estimated Bulk Price ($/lb) |
|---|---|---|---|
| Imbiber Beads IMB230301-R | Organic polymer sorbent composed of white 200- to 400-μm spherical beads | 0.64 | $9.50/lb (Source: vendor on 2/26/03) |
| Nochar A610 Petrobond | Organic polymer with irregular shaped particles that clump together | 0.29 | $6.65/lb (Source: vendor on 2/28/03) |
| Petroset II | Light brown to gray modified clay powder | 0.67 | $1.75/lb (Source: vendor on 2/26/03) |
| Petroset II-G | Granular light brown and gray clay particles | 0.77 | $1.70/lb (Source: vendor on 2/26/03) |

## BENCH-SCALE EXPERIMENTAL ACTIVITIES

The objective of this experimental work was to identify a sorbent capable of solidifying the PUREX waste that is cost effective and compatible with constituents in the PUREX waste and solidification processing equipment. The sorbent/surrogate combination must also remain stable under conditions that may be encountered during storage/shipment to an appropriate disposal facility.

The sorbents being investigated at the bench-scale were subjected to the following:

- determination of sorbent capacity; and
- sorbent property/mixing verification tests.

### Bench-Scale Sorbent Capacity Experimental Results

The objective of the bench-scale sorbent capacity experiments was to determine maximum sorbent capacity of each sorbent selected for testing when combined with the organic PUREX surrogate. Saturated paste extracts were made using the sorbent material and the organic PUREX surrogate at several loading rates. The resulting pastes were allowed to stand covered overnight and then subjected to a paint filter test to ensure that free liquids were not present. The sample with the greatest amount of organic PUREX that passed the paint filter test was used to calculate 100% or maximum sorbent capacity. A method combining water-/soil-saturated paste extracts and SW-846 Method 9095A, Paint Filter Liquids Test, was used as a basis for the procedure (3,4). Testing was performed at room temperature. Observations made during the surrogate/sorbent combinations preparation included consistency of each sorbent/PUREX combination, volumetric expansion of the sorbent/surrogate combinations, dust production potential associated with each sorbent, and any evidence of reaction between the surrogate and sorbent (i.e., precipitation of solids, fuming, fizzing, heat evolution, etc.). The data for the sorbent capacity tests using the organic PUREX surrogate are summarized in Table IV. Because the densities of the sorbents tested vary, the sorbent capacities are reported on a weight-of-sorbent to weight-of-PUREX basis with associated ratios, a weight-of-sorbent to volume-of-PUREX basis, and a volume-of-sorbent to volume-of-PUREX basis with associated ratios.

### Bench-Scale Maximum Sorbent Capacity Results

All four sorbent materials had relatively high sorbent capacities on a volumetric and weight basis. Imbiber Beads IMB230301-R had the best sorbent capacity on a volumetric and weight basis. Petroset II and Petroset II-G provided midrange sorbent capacities on a volumetric and weight basis. Nochar A610 Petrobond had a midrange capacity similar to the Petroset products on a weight basis, but the low density of the material adversely affected the volumetric capacity. This could negatively impact Nochar A610 Petrobond in comparison to the other materials if there is a limited volume available for sorbent in this particular application.

### Volumetric Expansion at Maximum Sorbent Capacity on a Bench-Scale

Volumetric expansion was calculated by dividing the final volume of the saturated paste by the volume of liquid added to create the saturated paste. These numbers should be considered approximate due to the crude graduations on the sample jars used, the difficulty in reading volumes of samples with irregular surfaces, and the visual interferences caused by paste adhering to the sides of the sample jars. All of the sorbents had limited volumetric

expansions ranging from 1.1 to 1.2 per milliliter of PUREX solidified when sorbents were applied at maximum capacity. If a safety factor of sorbent was applied, larger volume increases would be expected to occur.

**Dust Production Potential Evaluation During Bench-Scale Tests**

Dust production potential was evaluated for each sorbent using visual observation as the sorbents were added to the sample jars in preparation for the saturated paste tests. Petroset II yielded a high amount of dust upon addition to the sample jars. Petroset II-G and Nochar A610 Petrobond created a minimal amount of dust. On first inspection, Nochar A610 Petrobond, with its fine particles, appeared to be a dust producer. However, the material clumps together to prevent production of dust. Petroset II-G was expected to have a greater level of dust potential at the bottom of the storage container due to settling of the smaller particles over time. There was no noticeable dust associated with the Imbiber Beads IMB230301-R. However, the small size of the particles, coupled with the electrostatic nature of the material, made it difficult to control the spread of this material.

**Supplemental Observations for Bench-Scale Tests**

Other observations noted during the sorbent capacity testing are given below.

- No reactions between the sorbents and the PUREX surrogate were evident.
- Petroset II was difficult to mix when excess sorbent was present. The consistency of the mixture was like peanut butter and adhered to the spatula, causing problems with quantitative transfers.
- At 100% capacity, both Petroset II products resulted in smooth gels; Imbiber Beads IMB230301-R had the appearance of gelled beads; and Nochar A610 Petrobond resulted in a hard, sticky gel when stored overnight.
- Several saturated pastes had to be prepared with each sorbent material before the 100% capacity was determined.

Table IV Summary of sorbent capacity experimental data

| **Sorbent Name** | **Sorbent Description** | **Sorbent Capacity (g sorbent/g PUREX)** | **Weight-Based Waste Loading Ratio (wt sorbent:wt PUREX)** | **Sorbent Capacity (g sorbent/mL PUREX)** | **Sorbent Capacity (mL sorbent/ mL PUREX)** | **Volume-Based Waste Loading Ratio (vol sorbent: vol PUREX)** | **Volumetric Expansion/ mL PUREX** | **Consistency of Mixture** | **Paint Filter Test Result** | **Dust Production Potential** |
|---|---|---|---|---|---|---|---|---|---|---|
| Imbiber Beads IMB230 301-R | Spherical, white beads | 0.12 | 1:8 | 0.10 | 0.15 | 1:7 | 1.1 | Gelled beads | Pass | No, but very electrostatic |
| Nochar A610 Petrobond | White, lightweight, clumpy powder | 0.19 | 1:5 | 0.16 | 0.54 | 1:2 | 1.2 | Hard, sticky gel | Pass | Minimal; while material is fine, it clumps together |
| Petroset II | Flour-like, tan powder | 0.17 | 1:6 | 0.14 | 0.21 | 1:5 | 1.1 | Smooth, silky gel | Pass | Yes |
| Petroset II-G | Granular, kitty litter-type, gray solid | 0.19 | 1:5 | 0.15 | 0.20 | 1:5 | 1.1 | Smooth gel | Pass | Minimal; greater dust at bottom of bag |

After the sorbent capacity experiments were completed, the sorbent property/mixing verification study was undertaken.

**Sorbent Property/Mixing Verification Experimental Results**

The objective of the sorbent property/mixing verification tests was to determine the behavior and stability of surrogate/sorbent combinations. The surrogate liquid/sorbent combinations for the sorbent property/mixing verification tests were prepared using a fixed amount of surrogate and adding an appropriate amount of sorbent to achieve 50% (twice as much sorbent by weight than was necessary to sorb a given volume of organic PUREX surrogate) and 100% saturation of the sorbent (i.e., maximum capacity). The data gathered during the sorbent capacity testing were used to determine the amount of sorbent added to achieve the appropriate percentage saturation. Six methods of combining the surrogate with the sorbent were evaluated. The six methods were:

- adding sorbent to PUREX surrogate in the reaction vessel with and without mixing;
- adding PUREX surrogate to sorbent in the reaction vessel with and without mixing; and
- adding PUREX surrogate and sorbent simultaneously to the reaction vessel with and without mixing.

Table V provides a matrix of the applicable tests performed for each sorbent.

Table V Test matrix for each sorbent

| **Test Condition** | 1 | | 2 | | 3 | | 4 | | 5 | | 6 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Addition Method** | Sorbent added to surrogate | | | | Surrogate added to sorbent | | | | Sorbent and surrogate added simultaneously | | | |
| **Mixing/No Mixing** | Mix | | No mix | | Mix | | No mix | | Mix | | No mix | |
| **% Saturation** | 100% | 50% | 100% | 50% | 100% | 50% | 100% | 50% | 100% | 50% | 100% | 50% |
| Notes:<br><br>1. Quantitative measurements include capacity, time to sorb surrogate present, and volumetric expansion.<br>2. Qualitative observations include evidence of reaction, long-term stability, and consistency of product. | | | | | | | | | | | | |

Observations made during the PUREX/sorbent combinations preparation included:

- time for the sorbent to soak up the liquid—sorption rate;
- any evidence of reaction between the surrogate and sorbent (i.e., fuming, fizzing, heat evolution, precipitation products, etc.);
- behavior of the surrogate/sorbent combinations over time;
- any loss of stability of the combinations over time (i.e., separation of surrogate from the sorbent);
- mixing behavior of the sorbent/surrogate combinations;
- consistency of the combinations; and
- volumetric expansion of the sorbent/surrogate combinations over time.

Data from the sorbent property/mixing verification study are summarized in Table VI.

Table VI Summary of sorbent property/mixing verification studies data

| Sorbent Name | Addition Method | Mixing? | % Capacity | Sorption Rate (mL/min.) | Consistency of Mixture | Volumetric Expansion/ mL PUREX | Paint Filter Test Result | Observations |
|---|---|---|---|---|---|---|---|---|
| Imbiber Beads IMB230301-R | Sorbent to PUREX | No | 100 | 11 | White, opaque gel at bottom with ice appearance on top of sample | 1.1 | Pass | Samples without mixing are more dense than those prepared with mixing |
| Imbiber Beads IMB230301-R | PUREX to sorbent | No | 100 | 10 | White, opaque gel at bottom with ice appearance on top of sample | 1.1 | Pass | -- |
| Imbiber Beads IMB230301-R | Simultaneous | No | 100 | 10 | White, opaque gel at bottom with ice appearance on top of sample | 1.2 | Pass | -- |
| Imbiber Beads IMB230301-R | Sorbent to PUREX | Yes | 100 | 9 | White, opaque gel with mud-like cracks on top surface of sample turning white at edges | 1.1 | Pass | Samples prepared with mixing contain entrained air |
| Imbiber Beads IMB230301-R | PUREX to sorbent | Yes | 100 | 9 | White, opaque gel with mud-like cracks on top surface of sample turning white at edges | 1.1 | Pass | -- |
| Imbiber Beads IMB230301-R | Simultaneous | Yes | 100 | 11 | White, opaque gel with mud-like cracks on top surface of sample turning white at edges | 1.2 | Pass | -- |
| Imbiber Beads IMB230301-R | Sorbent to PUREX | No | 50 | 29 | Icy, white beads with opaque streaks throughout | 1.7 | Pass | -- |
| Imbiber Beads IMB230301-R | PUREX to sorbent | No | 50 | 27 | Icy, white beads with opaque streaks throughout | 1.4 | Pass | -- |
| Imbiber Beads IMB230301-R | Simultaneous | No | 50 | 27 | Icy, white beads with opaque streaks throughout | 1.8 | Pass | -- |
| Imbiber Beads IMB230301-R | Sorbent to PUREX | Yes | 50 | 24 | White, opaque gel at bottom with ice appearance on top of sample | 1.4 | Pass | -- |
| Imbiber Beads IMB230301-R | PUREX to sorbent | Yes | 50 | 24 | White, opaque gel at bottom with ice appearance on top of sample | 1.5 | Pass | -- |
| Imbiber Beads IMB230301-R | Simultaneous | Yes | 50 | 23 | White, opaque gel at bottom with ice appearance on top of sample | 1.6 | Pass | -- |
| Petroset II-G | Sorbent to PUREX | No | 100 | 0.4 | Soft gel | 1.2 | Pass | Samples prepared without mixing are denser than the mixed samples |
| Petroset II-G | PUREX to sorbent | No | 100 | 0.5 | Soft gel | 1.2 | Pass | -- |
| Petroset II-G | Simultaneous | No | 100 | 0.4 | Soft gel | 1.2 | Pass | -- |
| Petroset II-G | Sorbent to PUREX | Yes | 100 | 0.02 | Soft gel | 1.2 | Pass | Free-liquid pools in indentations in samples |
| Petroset II-G | PUREX to sorbent | Yes | 100 | 0.02 | Soft gel | 1.2 | Pass | Free-liquid pools in indentations in samples |
| Petroset II-G | Simultaneous | Yes | 100 | 0.02 | Soft gel | 1.2 | Pass | Free-liquid pools in indentations in samples |
| Petroset II-G | Sorbent to PUREX | No | 50 | 1.3 | Peanut butter consistency gel | 1.2 | Pass | -- |

| Sorbent Name | Addition Method | Mixing? | % Capacity | Sorption Rate (mL/min.) | Consistency of Mixture | Volumetric Expansion/ mL PUREX | Paint Filter Test Result | Observations |
|---|---|---|---|---|---|---|---|---|
| Petroset II-G | PUREX to sorbent | No | 50 | 1.4 | Peanut butter consistency gel | 1.2 | Pass | -- |
| Petroset II-G | Simultaneous | No | 50 | 1.2 | Peanut butter consistency gel | 1.2 | Pass | -- |
| Petroset II-G | Sorbent to PUREX | Yes | 50 | 1.0 | Hard, peanut butter consistency paste | 1.2 | Pass | -- |
| Petroset II-G | PUREX to sorbent | Yes | 50 | 1.0 | Hard, peanut butter consistency paste | 1.2 | Pass | -- |
| Petroset II-G | Simultaneous | Yes | 50 | 1.1 | Hard, peanut butter consistency paste | 1.2 | Pass | -- |
| Nochar A610 Petrobond | Sorbent to PUREX | No | 100 | 0.02 | Plastic, flowable, medium, putty-like gel | 1.2 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | PUREX to sorbent | No | 100 | 0.02 | Plastic, flowable, medium, putty-like gel | 1.2 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | Simultaneous | No | 100 | 0.02 | Plastic, flowable, medium, putty-like gel | 1.2 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | Sorbent to PUREX | Yes | 100 | 25 | Plastic, flowable, medium, putty-like gel | 1.3 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | PUREX to sorbent | Yes | 100 | 25 | Plastic, flowable, medium, putty-like gel | 1.4 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | Simultaneous | Yes | 100 | 25 | Plastic, flowable, medium, putty-like gel | 1.3 | Pass | Samples start opaque white, then become clear with bubbles, and finally, clear with almost no bubbles or no bubbles |
| Nochar A610 Petrobond | Sorbent to PUREX | No | 50 | 3,000 | Hard, rubbery gel with extra sorbent powder on top—not flowable | 1.8 | Pass | Had to cut sample with spatula to get it out of the container |
| Nochar A610 Petrobond | PUREX to sorbent | No | 50 | 3,000 | Hard, rubbery gel | 1.8 | Pass | Had to cut sample with spatula to get it out of the container |
| Nochar A610 Petrobond | Simultaneous | No | 50 | 3,000 | Hard, rubbery gel with white on top | 1.8 | Pass | Had to cut sample with spatula to get it out of the container |
| Nochar A610 Petrobond | Sorbent to PUREX | Yes | 50 | 67 | Hard, rubbery, clumpy gel; some opaque parts; some clear parts | 2.0 | Pass | Had to cut sample with spatula to get it out of the container |
| Nochar A610 Petrobond | PUREX to sorbent | Yes | 50 | 50 | Hard, rubbery, clumpy gel; some opaque parts; some clear parts | 2.0 | Pass | Had to cut sample with spatula to get it out of the container |
| Nochar A610 Petrobond | Simultaneous | Yes | 50 | 50 | Hard, rubbery, clumpy gel; some opaque parts; some clear parts | 2.0 | Pass | Had to cut sample with spatula to get it out of the container |
| Petroset II | Sorbent to PUREX | No | 100 | Never sorbed liquid | Very soft gel | 1.2 | Fail | Free-liquid pools in indentation made in sample |

| Sorbent Name | Addition Method | Mixing? | % Capacity | Sorption Rate (mL/min.) | Consistency of Mixture | Volumetric Expansion/ mL PUREX | Paint Filter Test Result | Observations |
|---|---|---|---|---|---|---|---|---|
| Petroset II | PUREX to sorbent | No | 100 | Never sorbed liquid | Soft gel | 1.2 | Fail | -- |
| Petroset II | Simultaneous | No | 100 | Never sorbed liquid | Soft gel | 1.2 | Fail | -- |
| Petroset II | Sorbent to PUREX | Yes | 100 | 17 | Soft gel | 1.02 | Fail | -- |
| Petroset II | PUREX to sorbent | Yes | 100 | 17 | Soft gel | 1.02 | Fail | Free-liquid pools in indentation made in sample |
| Petroset II | Simultaneous | Yes | 100 | 17 | Soft gel | 1.0 | Fail | -- |
| Petroset II | Sorbent to PUREX | No | 50 | Time not recorded | Medium paste with sorbed powder on top | 1.4 | Pass | Sample cracked after 2 days |
| Petroset II | PUREX to sorbent | No | 50 | Time not recorded | Smooth, medium gel | 1.4 | Pass | Big crack in sample after 9 days |
| Petroset II | Simultaneous | No | 50 | Time not recorded | Medium paste with sorbed powder on top | 1.2 | Pass | -- |
| Petroset II | Sorbent to PUREX | Yes | 50 | 25 | Medium to hard paste with peanut butter consistency | 1.2 | Pass | -- |
| Petroset II | PUREX to sorbent | Yes | 50 | 17 | Medium to hard paste with peanut butter consistency | 1.2 | Pass | -- |
| Petroset II | Simultaneous | Yes | 50 | 33 | Medium to hard paste with peanut butter consistency | 1.2 | Pass | -- |

**Sorption Rate at 50% and 100% Capacity**

The sorption rate was determined by dividing the volume of free-liquid surrogate by the amount of time necessary to absorb this volume. Mixing time was included in this calculation. As expected, the sorption rate increased as more sorbent was added (50% capacity samples had higher sorption rates than samples at 100% capacity). The order of addition of the PUREX and sorbent had very little impact on the sorption rate for any of the sorbents. Nochar A610 Petrobond at 50% capacity had the highest sorption rates with and without mixing. Nochar A610 Petrobond at 50% with no mixing almost instantaneously sorbed the PUREX; however, the excess dry material may not be desirable for this application. At 100% capacity, mixing had a significant positive impact on sorption rate for Nochar A610 Petrobond. Mixing had very little impact on sorption rate for Imbiber Beads IMB230301-R. Petroset II-G had the lowest sorption rates (with or without mixing) compared to the polymer sorbents. Additional sorbent would likely increase the sorption rate for Petroset II-G. Petroset II did not sorb all of the free liquid in the 100% samples made without mixing during the test period; therefore, a sorption rate could not be determined.

**Consistency of Mixture at 50% and 100% Capacity**

The consistency of mixture data presented in Table VI represent the consistency of the mixture at the end of the 14-day testing period. Imbiber Beads IMB230301-R yield a white, opaque gel with excess sorbent on top when not mixed. If mixed, the final consistency of the mixture for PUREX and Imbiber Beads IMB230301-R is a white, opaque gel with cracks on the surface at 100% capacity and a white, opaque gel covered with loose, partially saturated beads on the top of the sample.

Nochar A610 Petrobond when added to PUREX (with or without mixing at 100% capacity) resulted in a plastic, flowable, medium, putty-like gel that was clear by the end of the testing period. At 50% capacity, the result was a hard, rubbery gel with extra sorbent on top when prepared without mixing and a hard, rubbery, clumpy gel when prepared with mixing.

Petroset II-G resulted in soft gels at 100% capacity regardless of the mixing scenario or addition order. At 50% capacity, the samples had a peanut butter consistency without mixing and a harder peanut butter consistency for the samples prepared with mixing.

Petroset II samples all failed the paint filter test at 100% capacity. The soft gel samples that were mixed did not have any evident free liquid but still failed the paint filter test. At 50% capacity, the Petroset II/PUREX samples were medium to hard pastes with peanut butter consistencies when mixed and pastes with extra partially saturated powder on top when not mixed. It is anticipated that the consistency of the Petroset II/PUREX mixtures would be detrimental during deployment due to adherence of the solidified product to mixing equipment, which would create equipment maintenance and contamination issues.

**Volumetric Expansion at 50% and 100% Sorbent Capacity**

Volumetric expansion was calculated by dividing the final volume of the saturated paste by the volume of liquid added to create the saturated paste. These numbers should be considered approximate due to the crude graduations on the sample jars used, the difficulty in reading volumes of samples with irregular surfaces, and the visual interferences caused by paste adhering to the sides of the sample jars. All of the sorbents had limited volumetric expansions ranging from 1.0 to 1.4 per milliliter of PUREX solidified at 100% capacity. At 50% capacity, volumes were larger (as expected) because twice as much sorbent was added. The Petroset II products maintained low volumetric expansions between 1.2 and 1.4 per milliliter of PUREX solidified. The Imbiber Beads IMB230301-R had lower volumetric expansion when mixed or when the PUREX was added to the sorbent without mixing (1.4 to 1.6) and higher volumetric expansion when not mixed and the sorbent was added to the PUREX or the PUREX and sorbent were added simultaneously, 1.7 and 1.8, respectively. Nochar A610 Petrobond had the highest volumetric expansions. Samples prepared without mixing had volumetric expansions of 1.8, and samples prepared with mixing had volumetric expansions of 2.0. It is anticipated that the optimum deployment strategy for Nochar A610 Petrobond lies between 50% and 100% capacity. Because less sorbent would be used in such a scenario, the volumetric expansion for Nochar A610 Petrobond would not be this high.

**Mixing Requirements for 50% and 100% Capacity**

Imbiber Beads IMB230301-R performed well in the mix and no-mix scenarios. Five-gallon tests should be performed using Imbiber Beads IMB230301-R with mixing and without mixing. Nochar A610 Petrobond should also be tested at a 5-gallon scale with mixing and no mixing (mixing enhances sorption rate). Petroset II-G does not require mixing—the product is formulated specifically for no-mix scenarios. Petroset II-G should be tested further with more sorbent to enhance the sorption rate. Petroset II, on the other hand, requires mixing to achieve efficient sorption rates.

**Supplemental Observations for Sorbent Property/Mixing Verification Study**

Other observations made during the sorbent property/mixing verifications studies are stated below.

- There was no evidence of any reactions between the sorbent materials and the PUREX surrogate.
- There was no loss in stability of any of the mixtures once the free liquid was sorbed.
- Imbiber Beads IMB230301-R are difficult to control and would present handling problems if deployed. This material is typically sold in pillows that provide a containment of the material; consequently, this is not an issue when using the product for its usual, intended use.
- Petroset II produced a lot of dust when added to the sample containers.

A summary of results from the bench-scale tests is presented in Table VII.

Table VII Summary of sorbent evaluation data

| Sorbent Name | Dust Production Potential | Optimum Deployment Strategy | Acceptable Final Wasteform Physical Properties? | Continue Testing at 5-Gallon Scale |
|---|---|---|---|---|
| Imbiber Beads IMB230301-R | No, but very electrostatic. Small spheres may present handling/safety issues | Use sorbent 1:4 (wt sorbent:wt PUREX) with either mixing or no mixing | Yes; although, when excess sorbent is used, the beads remain as beads and swell upon absorption, which may be less desirable than a more cohesive final wasteform | Yes; however, handling issues associated with this material coupled with its electrostatic nature may make this material undesirable for this application |
| Nochar A610 Petrobond | Minimal; while material is fine, it clumps together | Use sorbent 1:4 (wt sorbent:wt PUREX) with mixing | Yes; it is anticipated that the final wasteform at the optimum deployment strategy will be acceptable | Yes |
| Petroset II-G | Minimal; greater dust at bottom of bag | Use sorbent at 1:1.3 (wt sorbent:wt PUREX) with no mixing | Yes; it is anticipated that the final wasteform at the optimum deployment strategy will be acceptable | Yes |
| Petroset II | Yes | Use sorbent at 1:1.5 (wt sorbent:wt PUREX) with mixing | Yes; however, the peanut butter consistency of this product would make it difficult to mix/prevent spread of contamination | No; the dust production potential coupled with mixing equipment contamination/maintenance problems make this material undesirable for this application |

**Pretest Optimization of Waste Loading Ratios In Preparation for 5-Gallon Tests**

Prior to performing the 5-gal tests, the waste loading ratios recommended in the MSE bench-scale report (5) were tested at a larger scale to determine if those ratios should be changed for the 5-gal tests. Selected ratios were chosen for wasteforms that passed Paint Filter Tests (PFT). The results are summarized in Table VIII.

Table VIII Optimized waste loading ratios for 5-gal tests

| Sorbent Name | Recommended Waste Loading Ratio Following Bench-Scale Testing (wt Sorbent:wt PUREX) | Optimized Waste Loading Ratio (wt Sorbent:wt PUREX) | Rationale |
|---|---|---|---|
| Petroset II-G | 1:1.3 | 1.5:1 or 2:1 | More sorbent was added to increase sorption rate |
| Imbiber Beads IMB230301 | 1:4 | 1:6 or 1:5 | Optimized the ratio to use less sorbent and positively impact cost analysis |
| Nochar A610 Petrobond | 1:4 | 1:4 | N/A |

The amount of Petroset II-G added was increased from the recommended ratio of 1:1.3 (weight of sorbent to weight of PUREX surrogate) to 1.5:1 and 2:1 to increase the sorption rate. The amount of Imbiber Beads was reduced from the recommended ratio of 1:4 to 1:5 to positively impact the cost of using this material in this application. The waste loading ratio for Nochar was unchanged at 1:4.

**5-GALLON EXPERIMENTAL ACTIVITIES**

The sorbents being investigated at a 5-gallon scale and 55-gallon scale were subjected to the following:

- optimization studies to verify waste loading ratios determined during the bench-scale study;
- 5-gal-scale tests at optimum waste loading ratios;
- ignitability of solidified wasteforms;
- thermal stability of solidified wasteforms using selected sorbent materials;
- 55-gal drum tests using selected sorbent materials with a simplified surrogate; and
- cost evaluation of sorbents to determine cost per 1,000 gal of PUREX waste treated.

**5-Gallon Test Results**

For 5-gal tests, sorbents were evaluated on behavior at optimized waste loading ratios with the organic PUREX surrogate determined prior to the 5-gallon testing. SW-846 Method 9095A, Paint Filter Free Liquids Tests and SW-846 Method 9096, Liquids Release Test Procedure were used to determine if free liquids existed in the final wasteforms (4).

The surrogate liquid/sorbent combinations for the sorbent property/mixing verification tests were prepared using 2 gal of organic PUREX surrogate and adding an appropriate amount of sorbent to achieve the desired waste loading ratios determined during the bench-scale studies and pretest optimization studies. The methods of addition used during the 5-gal tests were determined from results of the bench-scale study, pretest optimization studies, and vendor recommendations.

Table IX summarizes the matrix of the applicable tests performed for each sorbent at the 5-gal scale.

Table IX Test matrix for each sorbent at a 5-gal scale

| **Sorbent Name** | **Weight-Based Waste Loading Ratio (wt Sorbent: wt PUREX)** | **Sorbent/Surrogate Addition Method** | **Mixing/ No Mixing** | **Rationale for Choosing Test Condition** |
|---|---|---|---|---|
| Petroset II-G (Shakedown) | 2:1 | Sorbent added to surrogate | No mixing | Results of bench-scale study and vendor recommendation |
| Petroset II-G | 2:1 | Sorbent added to surrogate | No mixing | Results of bench-scale study and vendor recommendation/duplicate test |
| Petroset II-G | 2:1 | Sorbent/surrogate added to container in phases | No mixing | Evaluate a phased approach to the solidification process |
| Petroset II-G | 2:1 | Sorbent added to surrogate | Mixing | Determine impact of mixing on solidification process |
| Petroset II-G | 2:1 | Sorbent added to surrogate at room temperature | No mixing | To determine impact of temperature on sorption rates |
| Petroset II-G | 1.5:1 | Sorbent added to surrogate at room temperature | No mixing | To determine impact of temperature on sorption rates/impact of using less sorbent |
| Nochar A610 Petrobond | 1:4 | Sorbent added to surrogate | Mixing | Results of bench-scale study and vendor recommendation |
| Nochar A610 Petrobond | 1:4 | Sorbent added to surrogate | No mixing | To determine impact of not mixing on sorbent performance |
| Nochar A610 Petrobond | 1:4 | Sorbent added to surrogate | Mixing | Results of bench-scale study and vendor recommendation/duplicate test |
| Nochar A610 Petrobond | 1:4 | Surrogate added to sorbent; no mix for 2 min, then mix | Mixing | To determine impact of delay in mixing to simulate potential upset conditions |
| Imbiber Beads IMB230301 | 1:6 | Sorbent added to surrogate | No mixing | To give indication whether sorbent material is difficult to control due to its electrostatic nature in larger quantities |
| Imbiber Beads IMB230301 | 1:5 | Surrogate added to sorbent | No mixing | To give indication whether this addition method is favorable for controlling the spread of sorbent material |
| Imbiber Beads IMB230301 | 1:5 | Sorbent added to surrogate | Mixing | To determine sorbent performance with mixing |
| Imbiber Beads IMB230301 | 1:5 | Sorbent added to surrogate | No mixing | Duplicate test |

Observations made during the PUREX/sorbent combinations preparation included:

- time for sorbent to soak up liquid;
- behavior of surrogate/sorbent combinations over time;
- any loss of stability of combinations over time (i.e., separation of surrogate from sorbent);
- mixing behavior of sorbent/surrogate combinations;
- consistency of final products;
- volumetric expansion of sorbent/surrogate combinations over time;
- ignitability of final wasteforms; and
- thermal stability of selected wasteforms.

The results of the 5-gal tests are summarized in Table X.

**Sorption Rate**

The sorption rate was determined by dividing the volume of PUREX liquid surrogate sorbed by the amount of time necessary to absorb this volume; the time required for mixing was included in this calculation. Mixing had a significant positive impact on the sorption rate for Nochar and Imbiber Beads. Nochar without mixing never sorbed the liquid, while with mixing, the sorption rate was the fastest for any sorbent at 2 gal/min. Imbiber Beads without

mixing had a sorption rate of 0.18 gal/min versus 0.8 gal/min when mixed. Petroset II-G had the slowest sorption rates with or without mixing, and mixing seemed to have a negative impact on sorption rate (0.05 gal/min when mixed compared with 0.13 with no mixing for samples prepared the same day). It is hypothesized that this finding could be the result of an interference at the surface of the sorbent while sorption or mixing is taking place, causing the creation of exposed sorption surfaces that would not have the modification agent available to enhance sorption. The sorption rate using less sorbent (1.5:1) had a much slower sorption rate (0.0002 gal/min) compared to a sorption rate of 0.09 gal/min at the 2:1 Petroset II-G sample prepared the same day. The 5-gal samples were stored outside at ambient temperature, and slower sorption rates were noted when the outside temperature was lower.

**Addition Sequence**

The addition sequence used to combine the surrogate and sorbents was varied for certain tests. The best addition sequence for all sorbents at a 5-gal scale was to add the full amount of sorbent to the liquid surrogate. When the surrogate was added to each of the dry sorbents without mixing, there were areas of unused sorbent in the 5-gal container. For Petroset II-G, a phased approach to addition was attempted. The Petroset II-G was added in three equal portions without mixing, which resulted in a nonuniform wasteform and slower sorption rate compared to samples where all of the sorbent was added at the same time.

**Consistency of Mixture**

The consistency of mixture data presented in Table X represents the consistency of the mixture at the end of the initial 14-day monitoring period. Imbiber Beads yielded a noncohesive wasteform of soft gelled beads when prepared with or without mixing. Representatives from SRS, who witnessed 5-gal testing on May 6, 2003, did not find the consistency of the final wasteforms for Imbiber Beads to be desirable for this application because the samples were not cohesive solidified wasteforms. Imbiber Beads were removed from further testing based on the consistency of the final wasteform.

Table X Summary of data from the 5-gal tests

| Date | Sorbent Name | Addition Method | Mixing? | Waste Loading Ratio (g Sorbent: g PUREX) | Sorption Rate gal PUREX/ min | Consistency of Mixture | Volumetric Expansion per mL of PUREX | PFT Results After 56 Days | LRT Results After 56 Days | Observations |
|---|---|---|---|---|---|---|---|---|---|---|
| 4/28/03 | Imbiber Beads | Sorbent into surrogate | No | 1:6 | 0.17 | Soft gelled beads | 1.13 | Pass | Fail | Final wasteform was not cohesive |
| 4/30/03 | Imbiber Beads | Sorbent into surrogate | Yes (1-min mix) | 1:5 | 0.80 | Soft gelled beads | 1.13 | Pass | Fail | Final wasteform was not cohesive; mixing enhanced sorption rate |
| 4/30/03 | Imbiber Beads | Surrogate into sorbent | No | 1:5 | 0.18 | Soft gelled beads—dry sorbent on top of sample | 1.13 | Pass | Fail | Addition of surrogate to sorbent did not use available sorbent |
| 5/6/03 | Imbiber Beads | Sorbent into surrogate | No | 1:5 | 0.19 | Soft gelled beads | 1.13 | Pass | Fail | -- |
| 4/28/03 | Nochar | Sorbent into surrogate | Yes (1-min mix) | 1:4 | 2 | Rubbery mixture | 1.25 | Pass | Fail | -- |
| 4/30/03 | Nochar | Sorbent into surrogate | No | 1:4 | Never sorbed all liquid | Dry sorbent layer on top, rubbery middle, viscous fluid on bottom | 1.63 | Fail | Fail | Performed better with mixing |
| 4/30/03 | Nochar | Sorbent into surrogate | Yes (1-min mix) | 1:4 | 2 | Rubbery mixture | 1.3 | Pass, funnel wet | Fail | -- |
| 5/6/03 | Nochar | Surrogate into sorbent | Yes (2-min no mix/1-min mix) | 1:4 | 0.62 | Rubbery mixture | 1.13 | Fail | Fail | -- |
| 4/24/03 | Petroset II-G (Shakedown) | Sorbent into surrogate | No | 2:1 | 0.4 | Hard paste | 2 | Pass | Pass | -- |
| 5/2/03 | Petroset II-G | Phased approach (sorbent added in three equal additions) | No | 2:1 | 0.08 | Hard paste | 2 | Pass | Pass | Adding sorbent in a phased approach was not desirable at this scale |
| 5/6/03 | Petroset II-G | Sorbent into surrogate | No | 2:1 | 0.13 | Hard paste | 2 | Pass | Pass | -- |
| 5/6/03 | Petroset II-G | Sorbent into surrogate | Yes (1-min mix, plus stirred after 30 min to sorb remaining surrogate) | 2:1 | 0.05 | Hard paste | 2 | Pass | Pass | without mixing |
| 6/10/03 | Petroset II-G | Sorbent into surrogate | No | 2:1 | 0.09 | Medium paste | 2.08 | Pass | Pass | -- |
| 6/10/03 | Petroset II-G | Sorbent into surrogate | No | 1:1.5 | 0.0002 | Hard paste | 1.75 | Pass | Fail | -- |

Nochar when added to PUREX with mixing resulted in a rubbery mixture. The unmixed Nochar samples had three distinct phases: (1) viscous liquid in the bottom of the bucket, (2) a rubbery mixture in the center, and (3) excess sorbent on top. During hot weather, some Nochar samples appeared to lose stability and certain areas of the sample wasteforms became a viscous syrup. This finding prompted the thermal stability tests that are discussed in detail later in this paper.

Petroset II-G samples were all characterized as hard pastes and were definitely the most cohesive wasteform for the waste loading ratios investigated. The hard nature of the wasteform made sampling for PFT and LRT difficult.

**Paint Filter and Liquid Release Test Results**

All samples passed the PFT after 56 days of storage, with the exception of two Nochar samples: one was an unmixed Nochar sample and the other was generated by adding the sorbent to the surrogate and waiting 2 min before mixing for 1 min. The mixture was much more difficult to homogenize when there was this delay before mixing was initiated.

The polymer (Nochar and Imbiber Beads) samples failed the LRT after 56 days of storage at the waste loading ratios tested. The LRT may not be the best test for determining the presence of free liquids for the polymer samples, which had very little structural strength. If LRT is the criteria for acceptance at the final storage site, additional sorbent would be required for Nochar and Imbiber Beads. A position paper from the Nevada Test Site (NTS) suggests other tests (oven dry test, shaker tests, and freeze-thaw tests) for solidified wasteforms (6). These tests were recommended for Nochar samples at 1:2, 1:3, and 1:4 to determine if free liquids are present after being subjected to conditions suggested by NTS. Wasteforms with additional sorbent were subjected to thermal stability tests and LRT for Nochar. The Imbiber Beads were not subjected to thermal stability tests because the noncohesive wasteform was not desirable for this application. Imbiber Beads were eliminated from further testing.

All Petroset II-G samples passed the PFT and LRT at 2:1. The sample at 1.5:1 waste loading ratio failed the LRT. Because the final Petroset II-G wasteforms had more strength than the polymer sorbents, they performed better when subjected to the more aggressive LRT.

**Volumetric Expansion**

Volumetric expansion was calculated by dividing the final volume of the sample in gallons by the volume of liquid sorbed (2 gal). The volumetric expansion was lowest for Imbiber Beads and was a constant at 1.13 regardless of addition method or whether the sample was mixed or not mixed. Volumetric expansion for Nochar ranged from 1.13 to 1.25 when the sorbent and surrogate were combined with mixing. When not mixed, the volumetric expansion of the sorbent/surrogate combination was 1.63, indicating a significant increase in volumetric expansion of the final wasteform when mixing is not employed. Petroset II-G samples had the highest volumetric expansions for the 5-gal tests ranging from 1.75 at a waste loading ratio of 1.5:1 up to 2.08 at the 2:1 waste loading ratio. Mixing did not have an impact on volumetric expansion for Petroset II-G.

**Mixing Requirements**

Imbiber Beads performed well in the mix and no-mix scenarios on a bench scale; however, at a 5-gal scale, mixing was required to achieve a homogenous wasteform. Nochar was also tested with mix and no-mix scenarios, and this product performed better when mixed. Petroset II-G does not require mixing; the product is formulated specifically for no-mix scenarios; however, both scenarios were tested. A no-mix scenario is superior for this product. Mixing had a negative impact on sorption rate for Petroset II-G, which could be due to an interference at the surface of the sorbent while sorption is taking place or creation of exposed surfaces that do not have the modification agent available to enhance sorption.

**Ignitability Results**

Samples for ignitability were collected from the 5-gal test containers. Samples were analyzed by Mountain States Analytical in Salt Lake City, Utah, according to SW-846 Method 1030, *Ignitability of Solids*. Two samples were collected from the 5-gal containers for each sorbent. The results are summarized in Table XI.

Table XI Ignitability results for selected 5-gal samples

| Sample Identification | Sample Result (mm/s) | U.S. Department of Transportation (DOT) Ignitability Requirement (mm/s) | Comments |
|---|---|---|---|
| Petroset II-G #1 no mix | < 0.17 mm/s | < 2.2 mm/s | Pass |
| Petroset II-G #2 no mix | < 0.17 mm/s | < 2.2 mm/s | Pass |
| Imbiber Beads (1:5 no mix) | 1.33 mm/s | < 2.2 mm/s | Pass DOT requirement, but sample did ignite |
| Imbiber Beads (1:5 with mixing) | 1.67 mm/s | < 2.2 mm/s | Pass DOT requirement, but sample did ignite |
| Nochar (1:4 with mixing) | < 0.17 mm/s | < 2.2 mm/s | Pass |
| Nochar (1:4 with mixing) | 1.59 mm/s | < 2.2 mm/s | Pass DOT requirement, but sample did ignite |

According to the U.S. Department of Transportation (DOT), samples with burn rates < 2.2 mm/s are considered nonignitable; therefore, all the samples passed this standard. Both Imbiber Beads samples ignited, and the flame traveled along the sample; however, this occurred below the DOT limit that would designate the material as ignitable. Neither of the Petroset II-G samples ignited. One of the Nochar samples ignited, and one did not. Higher ratios of Nochar will probably not ignite because the material has fire prevention properties. After the long-term stability of samples from the temporal/thermal stability tests is determined, the Nochar samples will be submitted to the laboratory for additional analyses that will provide additional data on the ignitability characteristics of Nochar at waste loading rates that use additional sorbent.

**Supplemental Observations for 5-gal Tests**

Other observations made during the 5-gal study are listed below.

- There was no evidence of any reactions between the sorbent materials and the PUREX surrogate.
- The Nochar sample prepared by adding the sorbent to the surrogate and waiting 2 min before mixing was very difficult to homogenize.
- The Nochar sample that was not mixed never became homogenous and had three distinct phases.
- Nochar samples stored outside and subjected to high temperatures of approximately 100 °F became unstable. Because of this finding, thermal/temporal stability tests were undertaken to investigate the stability of Nochar at various ratios using additional sorbent and Petroset II-G at various temperature conditions.

After the 5-gal tests were completed, all data collected to date were reviewed with SRS. Imbiber Beads were removed from further consideration due to the noncohesive nature of the final wasteform; only Petroset II-G and Nochar were subjected to thermal/temporal stability tests and 55-gal drum tests.

**THERMAL STABILITY STUDY RESULTS**

Based on preliminary results of the 5-gal tests, Nochar and Petroset II-G were subjected to thermal stability tests. Petroset II-G was subjected to thermal stability tests at 2:1, and Nochar was tested at waste loading ratios of 1:2, 1:3, 1:4, and 1:5. Additional waste loading ratios of Nochar were tested because instability of the 5-gal samples was noted after being exposed to higher storage temperatures during the summer of 2003 and because 5-gal samples at 1:4 had failed the LRT. Imbiber Beads were not subjected to thermal stability tests because the nature of the final wasteform is not desirable for this application based on 5-gal test results. Thermal stability samples were created in triplicate and stored at high (120 °F), low (35 °F), and ambient temperature, and the final set of samples was subjected to temperature cycling (1 day at 120 °F, 1 day at room temperature, and 1 day at 35 °F, etc.). Thermal

stability samples were monitored for 14 days. One sample from each set was retained in the storage condition to assess long-term stability of the wasteforms at a variety of storage temperatures.

The results of the thermal stability tests are summarized in Table XII.

**Mixing Requirements During Thermal Stability Study**

Petroset II-G samples were not mixed. All Nochar samples were mixed, and the addition of more sorbent to obtain the desired waste loading ratio had an impact on mixing requirements. As more sorbent was added to achieve waste loading ratios of 1:3 and 1:2, mixing became more difficult. The samples prepared at 1:2 with Nochar were very difficult to mix within the jars used for testing. Since this testing was completed, it has been discovered that at 1:2, it is best to add the surrogate to dry sorbent that has been premixed to break up sorbent clumps and maximize the surface area of sorbent in contact with the surrogate.

**Sorption Rate During Thermal Stability Study**

Petroset II-G samples again had relatively slow sorption rates compared to the Nochar samples. Petroset II-G sorption rates ranged from 21.3 to 37.0 mL/min with an average sorption rate of 30 mL/min. Nochar samples had varying sorption rates because the time to mix the samples was included in the sorption time. All samples, regardless of the waste loading ratio, had sorbed the surrogate by the end of the mixing cycle. The duration of the mixing cycle was determined by the time necessary to achieve a homogenous wasteform. At 1:5 and 1:4 waste loading ratios, the Nochar samples only required 10 s of mixing, which translates to a sorption rate of 1,080 mL/min. At a 1:3 waste loading ratio, 30 s of mixing was required, which gives a sorption rate of 361 mL/min. Samples for a 1:2 waste loading ration required 60 s of mixing, and the sorption rate was 181 mL/min. There was difficulty mixing the 1:2 Nochar samples in the jars selected for testing because there was no additional volume in the jars. Additional volume should be available in the final mixing container to allow complete homogenization of the sorbent and liquid waste.

**Consistency of Mixture During Thermal Stability Study**

After 14 days of storage, the consistency of the mixture in each container was noted. For Petroset II-G samples (regardless of storage condition), the consistency of the mixture was a hard paste. The samples that were rotated each day (1 day in the oven, next day at ambient, following day in the refrigerator, next day at ambient, etc.) were significantly harder following refrigeration compared with samples stored in the refrigerator for the entire test period.

The consistency of Nochar samples prepared at waste loading ratios of 1:5 and 1:4 that were rotated varied based on storage condition: after oven storage, the consistency was a thick flowable clear liquid, and after storage at ambient temperature and in the refrigerator, the consistency was a thick clear mixture. This is more evidence that Nochar exhibits instability at high temperatures for 1:5 and 1:4 waste loading ratios. At 1:3, Nochar samples were all white opaque rubbery solids on top of clear gels, and at 1:2 the samples were white opaque rubbery solids on top of clear gels with excess sorbent on top of the sample, indicating that these samples were thermally stable at waste loading ratios of 1:3 and 1:2. More vigorous mixing at 1:2 may be required to make the wasteform more homogenous by incorporating excess sorbent into the wasteform.

It was evident from visually inspecting the wasteforms that samples stored at different temperatures have slightly different characteristics: Nochar samples stored in the oven are more opaque than those stored at room temperature, Nochar samples stored in the refrigerator have more residual bubbles than those stored at room temperature, and rotating samples most closely resemble the samples subjected to oven storage.

Table XII Summary of data from the thermal/temporal stability tests

| Date | Sorbent Name | Storage Condition | Mixing? | Waste Loading Ratio (g Sorbent: g PUREX) | Sorption Rate (mL PUREX/min) | Consistency of Mixture After 14 Days | Volumetric Expansion per mL of PUREX | PFT Results After 14 Days | LRT Results After 14 Days | Observations |
|---|---|---|---|---|---|---|---|---|---|---|
| 8/4/03 | Petroset II-G-A | Rotating (1 day ambient, 1 day oven, 1 day ambient, 1 day refrigerator) | No | 2:1 | 27.2 | Hard paste | 2.3 | Pass | Pass | Rotating samples were much harder than other Petroset II-G samples stored at constant high, ambient, or low temperatures |
| 8/4/03 | Petroset II-G-B | Rotating | No | 2:1 | 31.2 | Hard paste | 2.3 | Pass | Pass | Rotating samples were much harder than other Petroset II-G samples stored at constant high, ambient, or low temperatures |
| 8/4/03 | Petroset II-G-C | Rotating | No | 2:1 | 37.0 | Hard paste | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Rotating samples were much harder than other Petroset II-G samples stored at constant high, ambient, or low temperatures |
| 8/4/03 | Petroset II-G-A | Oven (120 °F) | No | 2:1 | 35.1 | Hard paste | 2.3 | Pass | Pass | -- |
| 8/4/03 | Petroset II-G-B | Oven (120 °F) | No | 2:1 | 32.9 | Hard paste | 2.3 | Pass | Pass | -- |
| 8/4/03 | Petroset II-G-C | Oven (120 °F) | No | 2:1 | 30.3 | Hard paste | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Petroset II-G-A | Ambient (75 °F) | No | 2:1 | 21.3 | Hard paste | 2.3 | Pass | Pass | -- |
| 8/4/03 | Petroset II-G-B | Ambient (75 °F) | No | 2:1 | 22.4 | Hard paste | 2.3 | Pass | Pass | -- |
| 8/4/03 | Petroset II-G-C | Ambient (75 °F) | No | 2:1 | 30.5 | Hard paste | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Petroset II-G-A | Refrigerator (34 °F) | No | 2:1 | 35.1 | Hard paste | 2.3 | Pass | Fail | Low temperatures must allow liquid release |
| 8/4/03 | Petroset II-G-B | Refrigerator (34 °F) | No | 2:1 | 28.8 | Hard paste | 2.3 | Pass | Fail | Low temperatures must allow liquid release |
| 8/4/03 | Petroset II-G-C | Refrigerator (34 °F) | No | 2:1 | 28.5 | Hard paste | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Rotating | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable opaque fluid after oven storage; thick opaque mixture after ambient storage; thick opaque mixture after refrigeration | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Rotating | Yes | 1:5 | 1,080 (10-s mix) | Thick opaque fluid after oven storage; thick opaque mixture after ambient storage; thick opaque mixture after refrigeration | 1.1 | Pass | Fail | -- |

| Date | Sorbent Name | Storage Condition | Mixing? | Waste Loading Ratio (g Sorbent: g PUREX) | Sorption Rate (mL PUREX/min) | Consistency of Mixture After 14 Days | Volumetric Expansion per mL of PUREX | PFT Results After 14 Days | LRT Results After 14 Days | Observations |
|---|---|---|---|---|---|---|---|---|---|---|
| 8/4/03 | Nochar-C | Rotating | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable opaque fluid after oven storage; thick opaque mixture after ambient storage; thick opaque mixture after refrigeration | 1.1 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 9/11/03 | Nochar-A | Rotating | Yes | 1:4 | 1,080 (10-s mix) | Thick clear fluid after oven storage; thick clear mixture after ambient storage; thick clear mixture after refrigeration | 1.2 | Pass | Fail | -- |
| 9/11/03 | Nochar-B | Rotating | Yes | 1:4 | 1,080 (10-s mix) | Thick clear fluid after oven storage; thick clear mixture after ambient storage; thick clear mixture after refrigeration | 1.2 | Pass | Fail | -- |
| 9/11/03 | Nochar-C | Rotating | Yes | 1:4 | 1,080 (10-s mix) | Thick clear fluid after oven storage; thick clear mixture after ambient storage; thick clear mixture after refrigeration | 1.2 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Rotating | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear gel | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Rotating | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear gel | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Rotating | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear gel | 1.4 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Rotating | Yes | 1:2 | Immediate | White clear rubbery solid with excess sorbent on top | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform; barely failed LRT |
| 8/4/03 | Nochar-B | Rotating | Yes | 1:2 | Immediate | White clear rubbery solid with excess sorbent on top | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform; barely failed LRT |
| 8/4/03 | Nochar-C | Rotating | Yes | 1:2 | Immediate | White clear rubbery solid with excess sorbent on top | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-A | Oven (120 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable opaque fluid | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Oven (120 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable opaque fluid | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Oven (120 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable opaque fluid | 1.1 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 9/11/03 | Nochar-A | Oven (120 °F) | Yes | 1:4 | 1,080 (10-s mix) | Clear solid with a few entrained bubbles | 1.2 | Pass | Fail | -- |
| 9/11/03 | Nochar-B | Oven (120 °F) | Yes | 1:4 | 1,080 (10-s mix) | Clear solid with a few entrained bubbles | 1.2 | Pass | Fail | -- |

| Date | Sorbent Name | Storage Condition | Mixing? | Waste Loading Ratio (g Sorbent: g PUREX) | Sorption Rate (mL PUREX/min) | Consistency of Mixture After 14 Days | Volumetric Expansion per mL of PUREX | PFT Results After 14 Days | LRT Results After 14 Days | Observations |
|---|---|---|---|---|---|---|---|---|---|---|
| 9/11/03 | Nochar-C | Oven (120 °F) | Yes | 1:4 | 1,080 (10-s mix) | Clear solid with a few entrained bubbles | 1.2 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Oven (120 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Oven (120 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Oven (120 °F) | Yes | 1:3 | 361 (30-s mix) | White sorbent solid on top of clear solid with entrained bubbles | 1.4 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Oven (120 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-B | Oven (120 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-C | Oven (120 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-A | Ambient (75 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable clear gel | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Ambient (75 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable clear gel | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Ambient (75 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick flowable clear gel | 1.1 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 9/11/03 | Nochar-A | Ambient (75 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with entrained bubbles | 1.2 | Pass | Fail | -- |
| 9/11/03 | Nochar-B | Ambient (75 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with entrained bubbles | 1.2 | Pass | Fail | -- |
| 9/11/03 | Nochar-C | Ambient (75 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with entrained bubbles | 1.2 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Ambient (75 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Ambient (75 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Ambient (75 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Ambient (75 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |

| Date | Sorbent Name | Storage Condition | Mixing? | Waste Loading Ratio (g Sorbent: g PUREX) | Sorption Rate (mL PUREX/min) | Consistency of Mixture After 14 Days | Volumetric Expansion per mL of PUREX | PFT Results After 14 Days | LRT Results After 14 Days | Observations |
|---|---|---|---|---|---|---|---|---|---|---|
| 8/4/03 | Nochar-B | Ambient (75 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-C | Ambient (75 °F) | Yes | 1:2 | 181 (60-s mix) | White sorbent on top of clear solid with entrained bubbles | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-A | Refrigerator (34 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-B | Refrigerator (34 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.1 | Pass | Fail | -- |
| 8/4/03 | Nochar-C | Refrigerator (34 °F) | Yes | 1:5 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.1 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 9/11/03 | Nochar-A | Refrigerator (34 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.2 | -- | -- | -- |
| 9/11/03 | Nochar-B | Refrigerator (34 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.2 | -- | -- | -- |
| 9/11/03 | Nochar-C | Refrigerator (34 °F) | Yes | 1:4 | 1,080 (10-s mix) | Thick clear gel with bubbles | 1.2 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | -- |
| 8/4/03 | Nochar-A | Refrigerator (34 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | Refrigerator samples had less clear portion than ambient and oven-stored samples |
| 8/4/03 | Nochar-B | Refrigerator (34 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | Pass | Fail | Refrigerator samples had less clear portion than ambient and oven-stored samples |
| 8/4/03 | Nochar-C | Refrigerator (34 °F) | Yes | 1:3 | 361 (30-s mix) | White opaque rubbery solid on top of clear solid with entrained bubbles | 1.4 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Refrigerator samples had less clear portion than ambient and oven-stored samples |
| 8/4/03 | Nochar-A | Refrigerator (34 °F) | Yes | 1:2 | 181 (60-s mix) | White opaque rubbery solid with excess sorbent on top | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-B | Refrigerator (34 °F) | Yes | 1:2 | 181 (60-s mix) | White opaque rubbery solid with excess sorbent on top | 2.3 | Pass | Fail | Sample difficult to mix and get homogenous wasteform |
| 8/4/03 | Nochar-C | Refrigerator (34 °F) | Yes | 1:2 | 181 (60-s mix) | White opaque rubbery solid with excess sorbent on top | 2.3 | N/A—retained to assess long-term stability | N/A—retained to assess long-term stability | Sample difficult to mix and get homogenous wasteform |

Note: All samples were prepared at ambient and allowed to sorb the PUREX; the samples were then placed in the appropriate storage conditions.

**Volumetric Expansion During Thermal Stability Study**

Volumetric expansion was calculated by dividing the final volume of the wasteform by the volume of PUREX solidified. Petroset II-G samples had a consistent volumetric expansion of 2.2. The Nochar volumetric expansion was impacted as more sorbent was added: at 1:5, the volumetric expansion was 1.1; at 1:4, the volumetric expansion was 1.2; at 1:3, the volumetric expansion was 1.4; and at 1:2, the volumetric expansion was 2.2. The volumetric expansion at 1:2 may be biased low because the jars were overflowing during mixing and the wasteforms were forced to fit in the volume of the jars used for testing.

**Paint Filter Test/Liquid Release Test Results During Thermal Stability Study**

Two of the three samples from each sample set were subjected to PFT. These two samples were then combined to perform the LRT. The third sample remains in storage to assess long-term stability.

All Nochar and Petroset II-G samples passed the PFT performed after 14 days of storage; therefore, all samples were subjected to an LRT. All Petroset II-G samples passed the LRT, with the exception of the Petroset II-G samples stored in the refrigerator. Low storage temperatures may interfere with the adsorption processes for Petroset II-G; however, no free liquid was visually evident in these samples. More testing is recommended to verify this finding to determine if Petroset II-G exhibits instability when stored at low temperatures.

All Nochar samples failed the LRT but came closer to passing the test as the amount of sorbent used increased.

**55-GAL TEST RESULTS**

Based on the results of the 5-gal test and thermal stability tests, an optimum deployment strategy for processing organic PUREX waste was devised at a 55-gal scale for Nochar and Petroset II-G. These two most promising sorbents were tested at the 55-gal scale using a simplified surrogate (tributyl phosphate and dyed kerosene). The purpose of the scaleup tests was to verify the sorbent-to-waste loadings determined in the 5-gal tests and determine the optimum process design for a full-scale system. A simple drum mixer was used for the 55-gal test using Nochar. The results of the 55-gal drum tests are summarized in Table XIII.

Table XIII Summary of results from 55-gal tests

| Sorbent Name | Waste Loading Ratio | Deployment Strategy | Sorption Rate (gal/min) | Volumetric Expansion | Final Wasteform Characteristics |
|---|---|---|---|---|---|
| Nochar | 1:2 | Add 20 gal of liquid surrogate to dry sorbent with mixing | Never sorbed | 1.9 | Rubbery mixture with pockets of viscous liquid with dry sorbent at bottom and sides of container |
| Petroset II-G | 2:1 | Add dry sorbent to 20 gal of liquid surrogate without mixing | 0.13 (sorbed 20 gal in 4 hr and 56 min) | 2.0 | Dry sorbent in center; hard paste at sides of container |

During the 55-gal drum tests, Petroset II-G performed similarly to how it performed during the 5-gal test. The dry sorbent was added to the PUREX surrogate without mixing, and the drum was monitored until all of the liquid (20 gal) was sorbed (approximately 4 hr 56 min). This translates to a sorption rate of 0.13 gal/min, which is comparable to the sorption rates calculated at the 5-gal scale. The final volume of the mixture was 40.4 gallons, which is approximately double the volume of PUREX surrogate sorbed.

PUREX surrogate was added to the Nochar with mixing. Mixing of the dry sorbent was ongoing prior to the addition of the PUREX. This strategy worked well at a 5-gal scale; however, Nochar never sorbed the free liquid and there were still unused pockets of Nochar in the 55-gal drum. While mixing was ongoing, a 3-inch-wide ring of unused sorbent was evident, indicating that the mixer used did not fully homogenize the drum contents. Better mixing will be necessary to homogenize the Nochar and PUREX surrogate. Nochar recommends the use of a paddle mixer to combine Nochar with the PUREX surrogate for future evaluations. The volume of the final Nochar wasteform was 38.7 gal or a volumetric expansion of 1.9.

**Cost Analysis Results**

The results from all tests to date were evaluated, and a cost evaluation was performed to project the cost ($/1,000 gal PUREX solidified) of solidifying PUREX using each sorbent. The cost evaluation data is summarized in Table XIV.

Table XIV Summary of sorbent evaluation data

| Sorbent Name | Optimum Waste Loading Ratio Based on All Testing to Date (wt Sorbent:wt PUREX) | Unit Sorbent Cost ($/lb) | Sorbent Cost ($/1,000 gal of PUREX) With Optimum Waste Loading Ratio |
|---|---|---|---|
| Imbiber Beads IMB230301-R | 1:5 | $9.50 | $13,200 |
| Nochar A610 Petrobond | 1:4 | $6.65 | $11,600 |
| Nochar A610 Petrobond | 1:3 | $6.65 | $15,400 |
| Nochar A610 Petrobond | 1:2 | $6.65 | $23,100 |
| Petroset II-G | 2:1 | $1.70 | $23,700 |

Petroset II-G and Nochar at a waste loading ratio of 1:2 are the most expensive options when sorbent cost alone is considered. Imbiber Beads are about one-half the cost of the Petroset II-G and Nochar materials at 1:2 despite its higher per unit sorbent costs. However, Imbiber Beads have been removed from consideration because the final wasteform was not cohesive. Mixing costs were not included in the cost evaluation. Nochar and Imbiber Beads would also have associated mixing costs. When the final metric for determination of free liquid is determined, may indicate that less Nochar will be necessary to achieve compliance. The impact of deploying Nochar at 1:3 and 1:4 would reduce the cost of deploying Nochar.

**CONCLUSIONS AND RECOMMENDATIONS**

The testing performed for SRS identified sorbents capable of solidifying PUREX organic waste surrogate. All of the sorbents solidified the PUREX organic waste surrogate; however, some of the properties of various sorbents were not desirable for this particular application. For example, Petroset II was eliminated from the evaluation process after bench-scale work due to sorbent dust production potential and sorbent handling concerns. Similarly, Imbiber Beads IMB230301-R were eliminated from consideration after the 5-gallon study because SRS decided that noncohesive nature of the final wasteform was not desirable for this application. A summary of the sorbent evaluation data from the 5-gallon and 55-gallon is presented in Table XV.

Table XV Summary of Phase II sorbent evaluation data

| Sorbent Name | Sorbent Cost ($/1,000 gal of PUREX) With Optimum Deployment Strategy | Final Wasteform Characteristics | Volume of Waste Generated per 1,000-gal of Waste Solidified | Thermal Stability/Presence of Free Liquid | Optimum Deployment Strategy for 55-gal drum[a] | Continue Testing at 55-gal or Larger Scale? |
|---|---|---|---|---|---|---|
| Imbiber Beads IMB230301-R at 1:2 | $13,200 | Soft gelled beads | 1,130 gal | No thermal stability tests performed; however, previous samples have passed PFT and failed LRT | Use sorbent 1:5 (wt sorbent:wt PUREX). Add sorbent to liquid waste with mixing | No; noncohesive nature of final wasteform is not desirable for this application |

| Sorbent Name | Sorbent Cost ($/1,000 gal of PUREX) With Optimum Deployment Strategy | Final Wasteform Characteristics | Volume of Waste Generated per 1,000-gal of Waste Solidified | Thermal Stability/Presence of Free Liquid | Optimum Deployment Strategy for 55-gal drum[a] | Continue Testing at 55-gal or Larger Scale? |
|---|---|---|---|---|---|---|
| Nochar A610 Petrobond at 1:4 | $11,600 | Thick clear gel with entrained bubbles | 1,200 gal | Thermal instability noted at 120 °F storage condition (mixture becomes flowable)<br><br>Pass PFT<br>Fail LRT at all temperature storage conditions | Use sorbent 1:4 (wt sorbent:wt PUREX). Add sorbent waste to liquid waste with mixing | No; thermal instability at high temperatures and failure of LRT make final wasteform undesirable |
| Nochar A610 Petrobond at 1:3 | $15,400 | White opaque rubbery solid on top of clear solid with entrained bubbles | 1,400 | Thermally stable from 34 °F to 120 °F (no movement/free liquid)<br><br>Pass PFT<br>Fail LRT at all temperature storage conditions | Use sorbent 1:3 (wt sorbent:wt PUREX). Add liquid waste to dry sorbent with mixing | Yes; however, samples failed LRT consistently at this ratio, but more in-depth mixing study is recommended if LRT is not the standard for determining presence of free liquids |
| Nochar A610 Petrobond at 1:2 | $23,100 | White sorbent on top of clear solid with entrained bubbles | 1,940 gal | Thermally stable from 34 °F to 120 °F (no movement/free liquid)<br><br>Pass PFT<br>Fail LRT at all temperature storage conditions | Use sorbent 1:2 (wt sorbent:wt PUREX). Add liquid waste to dry sorbent with vigorous mixing | Yes; however, final wasteform may not be desirable and the samples slightly failed LRT consistently at this ratio and homogenizing the wasteform with mixing used was difficult |
| Petroset II-G at 2:1 | $23,700 | Hard paste | 2,020 gal | Thermally stable from 34 °F to 120 °F (no movement/free liquid)<br><br>Pass PFT<br>Pass LRT<br>Fail LRT after refrigerator storage | Use sorbent at 2:1 (wt sorbent:wt PUREX). Add sorbent to liquid waste with no mixing | Yes; however, samples became less stable at low temperatures and failed LRT |

[a] The optimum deployment strategy listed in this table represents a best guess of the waste loading ratio at which each sorbent would be deployed if a 55-gal drum was the mixing/storage container.

The optimum deployment strategy listed in Table XV represents a best guess of the waste loading ratio at which each sorbent would be deployed given the information available. Further testing is recommended to ensure strategies are feasible for final deployment.

Imbiber Beads appear to be very attractive based on cost and volumetric expansion data; however, this sorbent was not recommended for this application due to the noncohesive nature of the final wasteform. Two sorbents are recommended to SRS for further consideration and evaluation were: (1) Petroset II-G and (2) Nochar. These were recommended because of their ability to absorb the PUREX surrogate and the characteristics of the final wasteforms from these two products. The recommendations given below should be taken into account regarding the deployment of these products.

- Nochar may require mixing for this application. The liquid waste should be added to the dry sorbent at a waste loading ratio of 1:2. At this waste loading ratio, the amount of sorbent is so large it was almost impossible to homogenize the mixture when the sorbent was added to the liquid surrogate. It is also advisable to mix the dry sorbent to break up clumps of sorbent to maximize the surface area available for sorption. Tests at a ratio of 1:3 should also be performed if LRT is not ultimately the standard to determine whether free liquid is present.

- If LRT is not the standard to determine whether free liquid is present, additional testing of Nochar at 1:2 and 1:3 and Petroset II-G will be necessary to determine compliance with the selected standard. Shaker tests and freeze-thaw tests may be appropriate in the upcoming phases of testing to ensure the stability of wasteforms under probable shipping/storage conditions and to comply with probable NTS requirements.
- Nochar is difficult to mix when there is any delay in starting mixing at 1:2 and 1:3 waste loading ratios (i.e., the mixture "cures" quickly). Any deployment scenario using Nochar would have to avoid delays in starting mixing. One strategy could be to begin mixing the dry sorbent prior to adding the waste so any mixing problems could be identified in advance of combining the liquid waste with the sorbent.
- When enough Nochar is added to make the final wasteform thermally stable at a 1:2 waste loading ratio, the final wasteform is very fluffy and has a high volume. The appearance of the final wasteform does not appear to be as homogenous as the ratios using less sorbent (1:4 and 1:3).
- Petroset II-G is formulated to remove the need for mixing. Petroset II-G should be added to the liquid waste in one application. Any deployment with Petroset II-G would not require mixing; however, the slow sorption rates would require enough time to be allotted so all the PUREX waste is sorbed.
- Petroset II-G has greater dust production potential than Nochar. Care should be taken to minimize dust production when using Petroset II-G.
- Additional 55-gal drum surrogate tests are recommended to test procedures generated for sorbent addition to drums or other deployment scenarios, including potential upset conditions. Also, a limited number of tests at 55 gal with the full surrogate are recommended to ensure that the simplified surrogate is representative of the behavior the sorbents with the full surrogate.
- If larger containers (B-12 and B-25 boxes) will be used for deployment, surrogate tests using these containers is recommended due to the differing geometry of a box versus a cylindrical 55-gal drum.
- The mixer tradeoff study findings/recommendations should be revisited to ensure the optimum mixing arrangement is deployed in future tests. Nochar recommends a paddle mixer for combining PUREX surrogate and Nochar A610 Petrobond.
- Offgas characterization tests should be carried out at temperatures other than ambient (such as 120 °F) and compared with the modeling results.
- Leachability testing should be performed using surrogates and actual waste.
- Further testing with actual waste at SRS is recommended once plausible deployment strategies are devised to ensure the waste loading ratios will generate a product that meets Resource Conservation and Recovery Act disposal requirements and waste acceptance criteria for disposal at SRS or NTS.

The evaluation of sorbents for solidification of organic PUREX waste from SRS indicates that solidification could provide a cost-effective alternative to incineration of this waste. If regulatory hurdles are overcome, deployment of solidification of PUREX waste could proceed.

## ONGOING ACTIVITIES

Imbiber Beads, Petroset II-G, and Nochar tests at a 5-gal scale continue to be monitored to determine the long-term stability of the final wasteforms. Offgas characterization tests and leachability tests involving Nochar and Petroset II-G are ongoing.

## ACKNOWLEDGEMENTS

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**REFERENCES**

1 Langton, C.A., Iversen, G.M., Fondeur, F., Creech, G.D., and Ogi, L.N., PUREX Organic Waste Solidification, Westinghouse Savannah River Company and Savannah River Technology Center, Aiken, SC, (December 2002).

2 Langton, C.A., PUREX Waste Solidification, Westinghouse Savannah River Company and Savannah River Technology Center, Aiken, SC, (November 2001).

3 Soil Science Society of America, "Saturation and Other Aqueous Extracts," Methods of Soil Analysis Part 3: Chemical Methods, pp. 419-422, (1996).

4 U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste—Physical/ Chemical Methods*, Method 1030—*Ignitability of Solids,* Method 8000B—*Determinative Chromatographic Separations* Method 9095A—*Paint Filter Liquids Test,* and Method 9096—*Liquid Release Test (LRT) Procedure*, EPA, Washington D.C., Office of Solid Waste web site: http://www.epa.gov/epaoswer/hazwaste/test/main.htm.

5 MSE Technology Applications, Inc., *Sorbent Testing for Solidification of Plutonium/Uranium Extraction Process Waste*, Prepared for DOE, March 2003.

6 Nevada Test Site, *Position Paper for High Moisture Content Waste,* Prepared by Nevada Test Site Generator Work Group—High Moisture Content Waste Subgroup, Revision 0, November 3, 1998.