### MERCURY REMOVAL FROM WASTE ORGANICS

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## ABSTRACT

Bench-scale mercury-removal scoping studies were performed on waste oil from the Savannah River Site and Oak Ridge National Laboratory (ORNL). Vacuum extraction as well as adsorption on sorbents was tested in small studies, and a field-scale sorption demonstration was later successfully completed on contaminated oil from ORNL. The field study was very successful, and the mercury in the oil was reduced to less than 0.2 mg/L (<0.25 mg/kg), from an initial concentration of 1.6 mg/kg. This technology is ready for further demonstration and treatment of U.S. Department of Energy waste oils.

## **INTRODUCTION**

The major scope of this work includes the use of Self-Assembled Mercaptan on Mesoporous Silica (SAMMS), developed at Pacific Northwest National Laboratory (PNNL), for treatment of mercury-contaminated oils (1, 2). Numerous U.S. Department of Energy (DOE) sites have radioactive organic liquids contaminated with mercury. Development of this application of the SAMMS material may provide the sites with a means of treating oil and possibly other organics.

### **METHODS**

Mercury-contaminated vacuum-pump waste oils were obtained from the Savannah River Site (SRS) and Oak Ridge National Laboratory (ORNL) waste management organizations. Vacuumextraction studies with the SRS oil were conducted in a radiological glove box because of the elevated levels of tritium in the oil. The potential for extraction of mercury from the oil was tested by applying a vacuum on a sample of the oil for a period of time, after which the headspace of the sample container was flushed with air. The method was evaluated at room and elevated temperatures.

The small-scale adsorption studies were carried out on SRS and ORNL oil in sealed containers with oil and various amounts of sorbent. The containers were mixed for different periods of time, and the oil was filtered to remove the sorbent prior to analyses. Several sorbents were tested.

The large-scale study was performed in a system consisting of a mixing container, an overhead mixer, a gear pump, a filter cartridge, and a receiving container. A variety of studies were performed using this setup, studying contact time and filter pore size.

# **RESULTS AND DISCUSSION**

### Vacuum-Extraction Tests

To investigate volatility of mercury species in the SRS oil, 2 mL of oil containing 52-84 mg/kg mercury was containerized and placed under vacuum (24 in. mercury) for 10 min at both room temperature and 70°C, after which the headspace was vented for 10 min. The treated oil contained 69.2 and 50.8 mg/kg mercury after treatment at room temperature and 70°C, respectively. It was concluded that the vacuum extraction performed did not have much (if any) effect on the removal of mercury species from the oil.

### **Sorption-Screening Tests**

The possibility of using selective sorbents for mercury removal was investigated using several sorbents [elemental sulfur; Mersorb LH (a sulfur-impregnated activated carbon), by Nucon International; Ionac SR-3, by Sybron Chemicals; Ionac SR-4; and SAMMS, produced by PNNL]. For each test, 0.2 g of sorbent was added to 3 mL of SRS oil and allowed to react for 48 h with intermittent shaking. The results may be seen in Fig. 1. As is noted, the SAMMS sorbent outperformed the others. This material has been described in detail in a report published by PNNL (1).



Fig. 1. Results from sorbent-screening test performed with SRS oil.

Additional mercury-sorption studies were performed with various amounts of SAMMS sorbent in 3-mL samples of SRS oil. The results are displayed in Fig. 2. As is noted, approximately 90% of the mercury was removed using the SAMMS sorbent at dosing rates of 0.2 to 0.3 g per 3 mL of oil.



Fig. 2. Effect of sorbent loading using SAMMS sorbent and SRS oil.

To determine the operating conditions for larger-scale implementation, treatability studies were performed on mercury-contaminated waste oil from ORNL. In these studies, several sorbents [SAMMS, granular activated carbon (GAC) by Calgon, Mersorb LH, and SIR-400 by ResinTech] and different sorbent dosings were investigated over a 24-h contact time with constant agitation. The raw waste oil contained 1.6 mg/kg mercury, and, unless noted, the samples were filtered through a 0.45-µm-pore-size filter prior to analyses (Fig. 3). The results showed that the SAMMS material was superior to other materials and that a dosing rate of 0.1 g of SAMMS per 50 mL of ORNL oil was sufficient to remove the mercury to below the target treatment goal of 0.2 mg/L (0.25 mg/kg). Since the ORNL oil contained a lower initial concentration of mercury than the SRS oil, it is not surprising that the decreased sorbent dosing rate used with this ORNL oil achieved a lower final concentration.



Fig. 3. Results from sorbent-screening and sorbent-loading experiments conducted with ORNL waste oil.

The necessary treatment time was studied by combining 2 g of SAMMS with 1 L of ORNL oil and taking periodic samples over a 24-h period, filtering each sample with a 0.45-µm-pore-size filter. It was shown that a contact time of only 1 h was sufficient to reduce the mercury to the target goal of 0.2 mg/L (0.25 mg/kg) for this particular application; however, the removal efficiency increased over the entire period. It should, however, be noted that other oils might not respond to this short treatment period, and thus it was decided to allow longer treatment times in subsequent experiments.

Since it was clear from previous experiments that filtration was an important part of the process, the filtration requirements were studied in a separate experiment. In this experiment, 1 L of ORNL oil was mixed with 2 g of SAMMS for 24 h, after which the mixture was pumped through an oil filter cartridge containing a commercial oil filter (nominal 10-µm pore size). The oil was then filtered using a variety of filter pore sizes (5, 1.2, 0.8, or 0.45 µm). The results indicated that a filter pore size of approximately 1 µm would be needed for effective separation of the SAMMS from the oil. These results compare well with published information about the relative size of SAMMS particles.

## **Sorption Field Test**

The work with ORNL oil culminated in the treatment of 16 L of waste oil using the 55-gal demonstration-scale system shown in Fig. 4. In two batch-treatment operations, 8 L of oil was combined with 17 g of SAMMS material, mixed for 20-22 h, and pumped through a filter cartridge with either a 0.5- or 1.0-µm-pore-size filter. The filtration of each batch of oil was completed in approximately 15 min, and the treated oil contained 0.149 and 0.048 mg/kg mercury, respectively, indicating that a 1.0-µm-pore-size filter was sufficient for the

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separation and actually outperformed the 0.5-µm filter. A possible explanation is that the pore size of these filters is nominal, not absolute. The treatment was successful, and the demonstration was completed on schedule as an ORNL performance milestone.



Fig. 4. Photograph of the mercury-in-oil removal process (MORP).

## CONCLUSIONS

Mercury was effectively removed from the oil via sorption using SAMMS. The method was demonstrated on a large scale using ORNL waste oil contaminated with mercury. This technology is ready for further demonstration and implementation when the SAMMS material is available in large quantities.

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