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Multi-Weight Isotherm Results for Mercury Removal in Upper East Fork Poplar Creek Water

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Chemical Technology Division

**MULTI-WEIGHT ISOTHERM RESULTS FOR MERCURY REMOVAL
IN UPPER EAST FORK POPLAR CREEK WATER**

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ABSTRACT

Many sorbents have been developed for the removal of mercury and heavy metals from waters; however, the majority of data published to date do not address the removal of mercury to the target levels represented in this project. The application to which these sorbents are targeted for use is the removal of mercury from microgram-per-liter levels to low nanogram-per-liter levels. Sorbents with thiouronium, thiol, amine, sulfur, and proprietary functional groups were selected for these studies.

The initial mercury content in the majority of the batch samples was significantly augmented so that the equilibrium concentration was similar to that found in the original stream sample for at least one sample.

Mercury was successfully removed from actual water via adsorption onto Ionac SR-4 (by Sybron Chemicals, Inc.), Keylex (by SolmeteX), and Mersorb (by Nucon International, Inc.) resins to levels below the target goal of 12 ng/L. A thiol-based resin (Ionac SR-4) performed the best, indicating that over 200,000 volumes of water could be treated with one volume of resin. The cost of the resin is approximately \$0.24 per 1000 gal of water.

1. PROJECT DESCRIPTION

The Engineering Development Section of the Chemical Technology Division at Oak Ridge National Laboratory (ORNL) will engage in several activities related to field and laboratory testing of sorbents and chelators for removal of mercury from water.

The work will combine demonstration of commercially (or near commercially) available ion-exchange or sorption resins. During batch (results presented here) and short kinetic studies, the best available commercial sorbent will be tested. Later, the best-performing and most cost-effective sorbents will be tested long term. Samples will be collected and analyzed for total mercury by an off-site laboratory.

2. WASTE STREAM INFORMATION

Two Department of Energy sites at ORNL and the Oak Ridge Y-12 Plant have large water streams with low concentrations of mercury. The Nonradiological Wastewater Treatment Plant (NRWTP) at ORNL is operated by waste management staff, and the Upper East Fork Poplar Creek (UEFPC) Characterization Area at the Y-12 Plant is managed by environmental restoration personnel. Both of these streams are regulated by National Pollutant Discharge Elimination System permits from the state of Tennessee. The flows are large — 350 gal/min (NRWTP) and 1700 gal/min (UEFPC) — and contain low levels of mercury in the range of 50 to 1000 ng/L. The desired treatment goal is 12 to 19 ng/L. The UEFPC receives water from various sources, some of which have already received limited mercury treatment; whether the surface water in the creek or waters bound for the creek will be treated in this study will be decided through discussion with environmental restoration staff at the Y-12 Plant. Some of the identified sources have flows in the range of 1 to 10 gal/min, with mercury concentrations of 2–40 µg/L. The NRWTP is currently using two activated carbon columns in series, each operating at a flow rate of 0.1 bed volumes (BV) per minute. The target level of 19 ng/L cannot be met with the current system, and a potential replacement sorbent is sought. The following results are from isotherm experiments conducted with UEFPC water (Outfall 200).

3. METHODS

3.1 MERCURY SORBENTS

Many sorbents have been developed for the removal of mercury and heavy metals from waters; however, the majority of data published to date do not address the removal of mercury to the target levels represented in this project. The application where these sorbents are targeted for use is in the removal of mercury from microgram-per-liter levels to low nanogram-per-liter levels. Sorbents with thiouronium, thiol, amine, sulfur, and proprietary functional groups were selected for these studies.

Ionac SR-3, by Sybron Chemicals, Inc., is a commercial macroporous polystyrene/divinylbenzene resin with (iso)thiouronium functional groups. This sorbent has been found to bind ionic mercury(II), methyl mercury, and elemental mercury and cannot be regenerated. It performs best at pH values between 0 and 6. At higher pH, the thiouronium group is converted into a thiol group. The cost of SR-3 is approximately \$12/L (SR-3). A similar resin is Purolite S-920 by the Purolite Company (\$24/L).

Ionac SR-4, by Sybron Chemicals, Inc., a macroporous, weakly acidic polystyrene/divinylbenzene cation resin with thiol functional groups, is commercially available at a cost of about \$14/L. It can be used at pH values ranging from 1 to 14. The recommended flow rate is 0.3 BV/min. Other materials with similar functionality are Duolite GT-73, by Rohm and Haas (\$35/L), and SAMMS (Self-Assembled Mesoporous Mercaptan Support), a newly developed inorganic thiol resin by Pacific Northwest National Laboratory (PNNL) that is not yet commercially available. The predicted cost is \$50/L. PNNL has presented data indicating that elemental and organic mercury may be adsorbed by SAMMS. In general, the thiol resins can sorb only mercury(II) ions.

Keyle:X, by SolmeteX, is polystyrene/divinylbenzene resin coated with a proprietary polymer with sulfur-based functional groups. Keyle:X is a mercury-specific resin that operates at high flow rates (0.2 to 2 BV/min). It is recommended that the water be pretreated with chlorine to 1–2 mg/L to ensure that the mercury is in the ionic state. The cost is \$126/L.

The Forager Sponge, by Dynaphore, Inc., is an open-cell cellulose sponge incorporating an amine-containing polymer that has selectivity for both cationic and anionic species of mercury and other heavy metals. It has the advantage of being a very porous compactible sponge; thus, column capacity can easily be adjusted by altering the packing density. The sponge can be pretreated with acid or base to change functionality (anionic versus cationic). The cost is \$5/L.

Mersorb LW, Mersorb 3, and Mersorb 1.5 mm (by Nucon International, Inc.) are commercial carbons impregnated with sulfur. The only difference between the three is particle size. Since the sorption is dependent upon precipitation of the sulfide, competing metal ions are those that form strong sulfide bonds. The cost is \$5/L.

3.2 EXPERIMENTAL PROCEDURE

The batch sorption experiment was conducted with water collected from East Fork Poplar Creek at Outfall 200 at the Y-12 Plant. The water was collected after a 1- μ m filter.

The batch isotherm samples were run in 250-mL I-Chem bottles with Teflon-lined closures. Sorbent masses were approximately 0.225, 0.45, 2.25, and 4.5 g; the solution volume was roughly 150 mL. The bottles were mixed on a Labquake® shaker for 24 h at room temperature (22 to 24°C). The aqueous phase was separated by using a Whatman Autovial syringe filter with a 0.45- μ m-pore membrane filter and transferred to a clean 125-mL I-Chem bottle prior to refrigeration, shipment, and analyses. The sample set included several quality control samples:

1. a process blank containing approximately 150 mL nanopure water (sample 8),
2. a stream sample that was not processed but was immediately refrigerated after collection (sample 9),
3. a 150-mL stream sample that had been fully processed but without sorbent (sample 7), and
4. a duplicate of a sorbent-treated stream sample (sample 3 is a duplicate of sample 2).

Additionally, loss of mercury to the glass walls of the sample containers was estimated by including three (samples 2, 7, and 11) of the original 250-mL sample bottles in the sample set. These three bottles were rinsed with a minimal amount of water to remove adhered sorbent; 50 mL nanopure water was then added to each bottle and shaken. No preservative was added to any of the samples before shipment. Analysis of mercury was carried out by Frontier Geosciences, Inc. The samples were oxidized with 1 vol % 0.2 N BrCl and analyzed by SnCl₂ reduction, dual gold amalgamation, and cold vapor atomic fluorescence detection.

Six samples in the batch sorption experiment were spiked with mercuric(II) nitrate to augment the total mercury content; two water samples were not spiked with mercury. The sorbents were used as received from the manufacturers, except in the case of Keylex, which was washed in nanopure water according to directions.

The sorption capacity was calculated using three different methods to account for various losses during the treatment procedure. In the first estimate of K_d (equal to sorption capacity divided by equilibrium concentration), the blank mercury level (sample 8) was subtracted from reported analytical results to calculate a net final mercury concentration in each of the treated samples. The initial mercury concentration was assumed to be the net mercury content of the unprocessed, refrigerated sample (sample 9), plus any spike added to the sample. The K_d for each treated sample was then calculated as follows:

$$K_d = \frac{([Hg]_9^{net} + [Hg]_i^{spike} - [Hg]_i^{net}) \times L_i}{[Hg]_i^{net} \times W_i}$$

In the second estimate of K_d , loss of mercury was attributed primarily to sorption on equipment walls. A sorption ratio for the "walls" was calculated by assuming that the initial net $[Hg]$ was represented by the untreated stream sample (sample 9) that was immediately refrigerated (plus any spike). The final $[Hg]$ was assumed to be that of the processed sample (plus spike) that did not contain sorbent (sample 7):

$$K_d^{walls} = \frac{([Hg]_9^{net} + [Hg]_7^{spike} - [Hg]_7^{net}) \times L_i}{[Hg]_7^{net}}$$

The K_d for each sample was then calculated as follows:

$$K_d = \frac{([Hg]_9^{net} + [Hg]_i^{spike} - [Hg]_i^{net}) \times L_i - K_d^{walls} \times [Hg]_i^{net}}{[Hg]_i^{net} \times W_i}$$

The third method to estimate K_d was to assume that each sample lost a fixed fraction of mercury as a result of volatilization and sorption onto equipment walls. The fractional loss was calculated based on the ratio of mercury concentration in the processed sample without sorbent (sample 7) to that of the refrigerated sample (sample 9) plus the amount of mercury initially added to the sample. The initial mercury concentration was then multiplied by the correction factor, cf , to determine K_d :

$$cf = \frac{[Hg]_7^{net}}{[Hg]_9^{net} + [Hg]_7^{spike}}$$

and

$$K_d = \frac{\{cf \times ([Hg]_s^{net} + [Hg]_i^{spike}) - [Hg]_i^{spike}\} \times L_i}{[Hg]_i^{net} \times W_i}$$

4. RESULTS

All process blanks (nanopure water) indicated a mercury content of <2 ng/L (see Appendix). This could be an existing contamination in the water or could come from the equipment used in the experiments. The initial mercury content in the majority of the batch samples was significantly augmented so that equilibrium concentration was well above the blank level and was similar to that found in the original stream sample for at least one sample. The three methods for calculating K_d gave almost identical results regardless of correction method used. It is also evident from the results that Ionac SR-4, Keyle:X, and Mersorb were able to remove mercury in the creek water to values below 12 ng/L (target goal).

The isotherm plots are shown in Figs. 1 through 5, in which the sorbent loading has been plotted as a function of the equilibrium concentration. The sorbent loading was calculated using the second correction method described above. Several results are evident. The experiment conducted with the thiuronium-based resin shows significant scatter in the data. One possible explanation may be that the sorbent partially broke down in the neutral water, producing a thiol-based resin. Some of the data in the low-concentration region appear scattered (see Figs. 2 and 5). Without additional experiments, it is difficult to determine which of the points are suspicious. The fact that the points do not seem to fall on the straight line may indicate that the form of the mercury in solution does not match that of the spiked solution. It is very possible that a fraction of the mercury is present in the elemental form (Hg^0), which may be converted to Hg^+ in the presence of larger amount of Hg^{2+} , which is found in the spiking solution. Ionic mercury is predominantly the sorptive species. This would explain why there was more mercury in solution at equilibrium in some unspiked experiments than in some spiked experiments.

Fig. 1. Isotherm results from multi-weight testing of Ionac SR-3, by Sybron Chemicals, Inc.

Fig. 2. Isotherm results from multi-weight testing of Ionac SR-4, by Sybron Chemicals, Inc.

Fig. 3. Isotherm results from multi-weight testing of Keyle:X, by SolmeteX.

Fig. 4. Isotherm results from multi-weight testing of the Forager Sponge, by Dynaphore, Inc.

Fig. 5. Isotherm results from multi-weight testing of Mersorb 1.5 mm, by Nucon International, Inc.

The results are summarized in Table 1 for treatment of a 1000-ng/L stream. As noted, the thiol-based resin (Ionac SR-4) performed much better than all others. The results indicate that 16,700 L of Outfall 200 water can be treated for \$1 in resin cost. This corresponds to a cost of approximately \$0.24/1000 gal water. The flow at Outfall 200 is approximately 2.5 million gallons per day; thus, the average daily cost for the resin would be about \$600.

5. CONCLUSIONS

Mercury was successfully removed from actual water via adsorption onto resins to levels below the target goal of 12 ng/L. A thiol-based resin (Ionac SR-4) performed the best, indicating that over 200,000 volumes of water could be treated with one volume of resin. The cost of the resin is approximately \$0.24 per 1000 gal of water.

Table 1. Summary results of isotherm testing

Sorbent	K_d (dry basis) ^a (L/kg)	K_d (bed basis) ^b (L/L _{BV})	K_d (cost basis) ^c (L/\$)
Sybron Chemicals, Inc. Ionac SR-3 (thiuronium)	50,000	26,700	2,200
Sybron Chemicals, Inc. Ionac SR-4 (thiol)	500,000 ^d	235,000	16,700
SolmeteX, Inc. Keyle:X (proprietary)	250,000	67,700	540
Dynaphore, Inc. Forager Sponge (amine)	3,000	270 ^e	54 ^e
Nucon International, Inc. Mersorb 1.5 mm	70,000	39,500	7,900

^aCalculated as sorbent capacity (ng mercury/kg dry sorbent) ÷ [1000 (ng mercury/L water)].

^bCalculated as [K_d (dry basis)] × (1 – moisture content) × [bulk density (kg/L)].

^cCalculated as [K_d (bed basis)] ÷ [cost (\$/L_{BV})].

^dExtrapolated value.

^eThese values can be increased by packing the Forager Sponge more tightly.

APPENDIX
DETAILS OF BATCH EXPERIMENTS

Table A- 1. Multi-weight sorption isotherm for Ionac SR-3, by Sybron Chemicals, Inc.

Sample bottle	Dry sorbent weight ^a (g)	Liquid volume (mL)	Mercury spike (ng/L)	Equilibrium concentration (ng/L) _{gross}	Equilibrium concentration (ng/L) _{net}
SR3-1B	0.1867	166.12	73,937	2,906	2,904.7
SR3-2B	0.3789	168.66	71,501	255	253.7
SR3-3B	0.3751	170.89	83,037	392	390.8
SR3-4B	0.3754	173.60	125,428	129	127.7
SR3-5B	1.8781	173.13	75458	246.3	245
SR3-6B	3.7529	170.38	68,323	32.29	30.99
SR3-7B	0	171.47	71,739	72,338	72,337
SR3-8B	0	168.92	0	1.30	0
SR3-9A	0	250	0	677.6	676.3
SR3-10B	0.1876	174.7	0	213.3	212
SR3-11B	0.3762	170.6	0	119.0	117.7
SR3-9A duplicate				665.2	663.9
SR3-9A spike (2,020 ng/L Hg added)				2,706.4	2,705.1
SR3-9A spike duplicate (2,020 ng/L Hg added)				2,797.1	2,795.8
SR3-2A bottle wash		50		32,214	
SR3-7A bottle wash		50		6,632.3	
SR3-11A bottle wash		50		844.4	

^aSorbent moisture content was 16.64%.

Table A- 2. Multi-weight sorption isotherm for Ionac SR-4, by Sybron Chemicals, Inc.

Sample bottle	Dry sorbent weight ^a (g)	Liquid volume (mL)	Mercury spike (ng/L)	Equilibrium concentration (ng/L) _{gross}	Equilibrium concentration (ng/L) _{net}
SR4-1B	0.1778	150.45	83,436	113	111.72
SR4-2B	0.3545	156.85	80,157	65.6	64.32
SR4-3B	0.3562	155.74	80,853	67.8	66.52
SR4-4B	0.3547	153.47	333,464	181	179.72
SR4-5B	1.7748	154.25	80,797	2.09	0.81
SR4-6B	3.5476	154.28	81,044	2.29	1.01
SR4-7B	0	148.70	84,149	77,900	77,898.72
SR4-8B	0	146.38	0	1.28	0
SR4-9A	0	150	0	754	752.72
SR4-10B	0.1771	151.33	0	8.86	7.58
SR4-11B	0.3549	146.92	0	5.98	4.7
SR4-9A duplicate				750	748.7
SR4-9A spike (2,000 ng/L Hg added)				2,030	2,028.7
SR4-9A spike duplicate (2,000 ng/L Hg added)				1,982	1,980.7
SR4-2A bottle wash		50		3,740	
SR4-7A bottle wash		50		12,900	
SR4-11A bottle wash		50		828	

^aSorbent moisture content was 26.85%.

Table A- 3. Multi-weight sorption isotherm for Keyle:X, by SolmeteX

Sample bottle	Dry sorbent weight ^a (g)	Liquid volume (mL)	Mercury spike (ng/L)	Equilibrium concentration (ng/L) _{gross}	Equilibrium concentration (ng/L) _{net}
KX-1B	0.1035	150.38	83,661	274.5	272.6
KX-2B	0.2078	157.17	80,388	248.7	246.8
KX-3B	0.2074	156.18	81,471	126.7	124.8
KX-4B	0.2079	153.77	333,726	1012	1010.1
KX-5B	1.0386	154.70	81,479	40.74	38.87
KX-6B	2.0777	153.89	83,312	17.74	15.87
KX-7B	0	148.54	84,234	74,544	74,542
KX-8B	0	146.83	0	1.87	0
KX-9A	0	150	0	793.1	791.2
KX-10B	0.1039	152.21	0	9.22	7.4
KX-11B	0.2081	147.16	0	7.83	5.96
KX-7B duplicate				7,2554	72,552.1
KX-7B spike (202,020 ng/L Hg added)				281,499	281,497.1
KX-7B spike duplicate (202,020 ng/L Hg added)				282,693	282,691.1
KX-2A bottle wash		50		13,504	
KX-7A bottle wash		50		7,693.1	
KX-11A bottle wash		50		267.8	

^aSorbent moisture content was 53.83%.

Table A- 4. Multi-weight sorption isotherm for the Forager Sponge, by Dynaphor, Inc.

Sample bottle	Dry sorbent weight ^a (g)	Liquid volume (mL)	Mercury spike (ng/L)	Equilibrium concentration (ng/L) _{gross}	Equilibrium concentration (ng/L) _{net}
Spg-1B	0.0292	316.23	46,549	40619	40,617.6
Spg-2B	0.0588	319.72	45,996	37674	37,672.6
Spg-3B	0.0584	318.15	49,357	39811	39,809.6
Spg-4B	0.0593	317.59	170,213	139,878	139,876.6
Spg-5B	0.2932	320.10	45,319	27,246	2,7244.59
Spg-6B	0.5889	320.41	46,776	24,488	24,486.59
Spg-7B	0	317.41	47,557	47,480	47479
Spg-8B	0	150	0	1.41	0
Spg-9A	0	150	0	805	803.6
Spg-10B	0.0296	322.14	0	509.3	507.9
Spg-11B	0.0589	317.22	0	596.9	595.49
Spg-9A duplicate				801.1	799.7
Spg-9A spike (2,020.2 ng/L Hg added)				2,874.6	2,873.2
Spg-9A spike duplicate (2,020.2 ng/L Hg added)				2,902.5	2,901.1
Spg-2A bottle wash		50		1,636.9	
Spg-7A bottle wash		50		7,084.1	
Spg-11A bottle wash		50		366.1	

^aSorbent moisture content was 53.83%.

Table A- 5. Multi-weight sorption isotherm for Mersorb 1.5 mm by, Nucon International, Inc.

Sample bottle	Dry sorbent weight ^a (g)	Liquid volume (mL)	Mercury spike (ng/L)	Equilibrium concentration (ng/L) _{gross}	Equilibrium concentration (ng/L) _{net}
MS-1B	0.2428	139.10	89432	914	913.2
MS-2B	0.4372	130.20	95545	424	423.2
MS-3B	0.4417	130.50	95326	392	391.2
MS-4B	0.4385	134.60	367831	735	734.2
MS-5B	2.1808	132.90	93604	2.97	2.19
MS-6B	4.3547	139.10	89432	1.81	1.03
MS-7B	0	139.90	88921	74081	74080
MS-8B	0	150	0	0.78	0
MS-9A	0	150	0	713	712.2
MS-10B	0.2468	141.00	0	7.05	6.3
MS-11B	0.4411	146.10	0	4.67	3.89
MS-9A spike (2020 ng/L Hg added)				2143	2142.2
MS-9A spike duplicate (2020 ng/L Hg added)				2136	2135.2
MS-2A bottle wash		50		111280	
MS-7A bottle wash		50		2497	
MS-11A bottle wash		50		382	

^aSorbent moisture content was 3%.

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