Biosorption of mercury from aqueous solutions using highly characterised peats

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SUMMARY

This research investigated the biosorption of mercury from aqueous solutions by six highly characterised peats. Samples of the peats were tested both in unaltered condition and after being treated with hydrochloric acid (HCl) to free up any occupied exchange sites. Other variables tested were sample dose, contact time, mixing temperature, and the concentration and pH of the mercury solution. Desorption studies were also performed, and tests were done to determine whether the peats could be re-used for mercury biosorption. The results indicate that all six peat types biosorb mercury from aqueous solutions extremely well (92−100 % removal) and that their mercury removal capacities are not significantly affected by manipulation of the various factors tested. The factor that had the greatest impact on the mercury removal capacities of the peats was the pH of the mercury solution. The optimal mercury solution pH for mercury removal was in the range 5−7 for four of the peats and in the range 2−3 for the other two. The desorption results indicate that it may be possible to recover up to 41 % of the removed mercury. All of the peat types tested can be repeatedly re-used for additional mercury biosorption cycles. Hence, their disposal should not become a hazardous waste problem.

KEY WORDS: cost-effective; environmentally friendly; heavy metals; remediation

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INTRODUCTION

Industrial activities are a major source of environmental contamination. Wastewater effluents containing heavy metals from industries such as metal plating, mining, oil refining, rubber processing, tanning and chemical manufacturing find their way into aqueous environments (Meena *et al.* 2008). The metals with the highest toxicities are lead, cadmium, chromium (VI) and mercury. Traditionally, heavy metal waste removal has been accomplished using physical and chemical techniques such as ion exchange and precipitation, reverse osmosis, electrodialysis, lime coagulation, and adsorption using activated carbon and lime ash. These techniques are not efficient because they are costly and have high energy input requirements. Metals may not be completely removed when these methods are applied, and toxic sludge can be generated (Krishnani *et al.* 2008). There is a need for cost-effective, environmentally friendly heavy metal remediation technologies.

An emerging technique known as biosorption has been presented as both effective and inexpensive in comparison to chemical and physical technology (Krishnani *et al.* 2008), and various biosorbent materials have been found to work well at removing heavy metals from aqueous solutions. Biosorption is the binding of metal ions and radionuclides onto the cellular structures of biological materials, including their ligands and functional groups. Biosorbent materials that are lignocellulosic (containing cellulose, hemicellulose and lignin) have high adsorption properties due to their ion exchange capabilities. Accordingly, they can be used as productive biosorbent material (Krishnani *et al.* 2008). It has been shown that biosorbent materials can be regenerated for re-use, and adsorbed metal ions can possibly be recovered from the biosorbent material (Volesky 2007). In addition, biosorbent materials are cost effective, efficient, do not require much energy input, and do not produce toxic sludge. The efficacy of biosorbent materials fluctuates when various factors such as pH, metal concentration, adsorbent dose, particle size and contact time are modified.

Peat is an abundant and inexpensive naturally occurring organic sediment derived from plants. Several studies have shown that peats are effective at removing heavy metals from aqueous solutions. Four papers present good reviews of the literature up to 2003 (McLellan & Rock 1986, Bailey *et al.* 1999, Brown *et al.* 2000, Babel & Kurniawan 2003), and additional relevant references include Chen *et al.* (2001) and Bulgariu *et al.* (2009b). Several studies including our own (Stack *et al.* 1994, Cohen & Stack 1995, Rizzuti *et al.* 1996, Rizzuti *et al.* 2003) have shown that the amounts and rates of metal extraction

can differ greatly between one type of peat and another. This is due to the fact that different peat types can vary widely in their botanical composition as well as in their chemical and physical properties. A few studies have looked at the mercury removal capacity of peat (Coupal & Lalancette 1976, Tummavuori & Aho 1980, Viraraghavan & Kapoor 1995, Bulgariu *et al.* 2008, Bulgariu *et al.* 2009a). In these studies the mercury removal capacities of only moss (e.g. *Sphagnum*) peats were tested.

The United States of America (USA) Environmental Protection Agency (EPA) has set the maximum contaminant level goal for mercury in drinking water at 2 ppb. The study reported here was funded by the Department of Environment (DOE) to develop cost-effective, environmentally friendly mercury remediation techniques, specifically in the context of waterborne mercury being discharged into the East Fork Poplar Creek from the Oak Ridge DOE site in Oak Ridge, Tennessee (USA). The purpose of this study was to determine the capacities of six different highly characterised peat types for biosorption of mercury from aqueous solutions at concentrations typical of those observed at Oak Ridge (within the range <1–40 ppb).

MATERIALS AND METHODS

Materials

The peats were obtained from the peat sample bank at the University of South Carolina. Six different peat types were used. They were chosen to represent a wide range of depositional environments and chemical and physical properties (Table 1).

The mercury (Hg^{2+}) solutions were prepared by diluting a 10 ppm stock solution (in 5 % nitric acid) to the desired concentrations in parts per billion (ppb) using ASTM Type II deionised water. The concentration of the initial working standard was 20 ppb.

All of the glassware used in this research was soaked in 10 % nitric acid for two hours and rinsed thoroughly with Type II deionised water. All sample tubes were soaked overnight in a 40 % HCl solution and rinsed with deionised water.

Mixing procedure

For the 'standard' procedure, 0.5 g of peat was weighed out, placed in a 250 ml flask, and 100 ml of 20 ppb mercury solution was added. The flask was then placed on a shaker (Thermo Scientific MaxQ 2000) at ~ 175 rpm to mix for 24 hours. In order to test the effects of different factors on the removal of mercury we varied the quantity of peat, the

concentration of the mercury solution, the mixing time and other factors. Details of the various treatments that were applied are given below.

Sample dose
To investigate the effect of sample dose, 0.5, 1.0 or 2.0 g of peat was mixed with 100 ml of 20 ppb (pH 3) mercury solution for 24 hours.

Contact time
To study the effect of contact time, 0.5 g of each peat type was mixed with 100 ml of 20 ppb (pH 3) mercury solution for 2, 24 or 48 hours.

Mixing temperature
To determine the effect of mixing temperature, 0.5 g of peat was mixed with 100 ml of 20 ppb (pH 3) mercury solution for 24 hours in a temperature-controlled shaker set at room temperature (24±1 °C), 30 °C, 35 °C or 40 °C.

Mercury solution concentration
To investigate the effect of mercury solution concentration, 0.5 g of each peat type was mixed with 100 ml of 5, 10, 20, 30 or 40 ppb mercury solution (pH 3) for 24 hours. These (low) concentrations were chosen to represent the waterborne mercury concentrations in the East Fork Poplar Creek.

pH of mercury solution
To test the effect of varying pH of the mercury solution, 0.5 g of each peat type was mixed with 100 ml of 20 ppb mercury solution at different pHs in the range 2−10 (increment unity) for 24 hours. This was achieved by adjusting the pH of the working mercury solution above or below that of the initial working standard solution (3.0 +/-0.1) using a 0.05N solution of sodium hydroxide and 0.01N hydrochloric acid.

HCl treatment
The purpose of the HCl treatment was to remove metal ions attached to the peat sample and, hence, free up sites to bond with mercury ions. Each sample was mixed with 10 % HCl for one hour. This step was repeated with fresh HCl for another hour. The sample was then washed with deionised water until a pH of approximately 6 was reached. Next, 0.5 g of the HCl-treated peat sample was mixed with 100 ml of a 20 ppb (pH 3) mercury solution for 24 hours.

Desorption of mercury
To determine whether the removed mercury could be recovered and recycled, peat samples that had been used for the biosorption of mercury were tested to

Table 1. Key characteristics of the peat samples used in this research (Cohen *et al.* 1991). ASTM: American Society for Testing and Materials (international standards organisation); FL: Florida; GA: Georgia.

sample code	MS	ON	LS	OT	RH	LN
sample name	Maine *Sphagnum*	Okefenokee *Nymphaea*	Loxahatchee Sawgrass	Okefenokee *Taxodium*	Shark River *Rhizophora*	Loxahatchee *Nymphaea*
location of origin	Maine	Okefenokee Swamp, GA	Loxahatchee WL Refuge, FL	Okefenokee Swamp, GA	Everglades National Park, FL	Loxahatchee WL Refuge, FL
dominant botanical components	*Sphagnum*	*Nymphaea, Sagittaria,* grass-sedge	Grass-sedge, *Nymphaea,* fern	*Taxodium, Persea*	*Rhizophora*	*Nymphaea, Sagittaria*
ASTM classification fiber (area-%)	Fibric (81)	Hemic (50)	Hemic (48)	Sapric (18)	Hemic (55)	Hemic (40)
Ash (wt-%)	0.8	12.4	7.2	12.8	31.0	6.4
C (wt-%)	52	52	55	51	36	54
Total aldehydes (%)	12.891	13.837	8.112	11.021	7.061	8.012
Total furans (%)	2.926	4.730	5.046	6.259	2.851	5.255
Total furanones (%)	15.068	4.592	4.363	3.376	5.720	7.791
Total pyranones (%)	7.478	9.143	1.551	3.416	5.701	5.250
Total other ketones (%)	3.548	2.753	2.949	3.329	1.578	4.842
Total guaiacyl lignins (%)	10.529	12.537	12.580	13.865	14.225	13.172
Total other lignins (%)	18.683	9.244	16.767	13.432	13.067	10.477
Humic acids content (%)	5.5	4.9	4.6	14.5	2.7	7.1
Fulvic acids content (%)	0.06	0.02	0.12	0.27	0.74	0.20
Lignin/cellulose ratio	0.74	1.15	1.58	1.41	1.57	1.35

determine the amount of mercury that could be desorbed from them afterwards. Peat that had already been used once for biosorption of mercury (0.5 g of sample mixed for 24 hours with 20 ppb mercury solution at pH 3) were mixed with 100 ml of a 10 % HCl solution for two hours.

Re-using samples for biosorption of mercury
Peat samples that had been used for biosorption of mercury were also tested to determine whether they could be re-used for this purpose and whether desorbing the mercury from the peat would improve its effectiveness when re-used. Samples were tested (with and without going through desorption) for their mercury removal capacities after being used once. Peat that had already been used once for biosorption of mercury (24 hours with 20 ppb mercury solution at pH 3) was mixed with 100 ml of fresh 20 ppb mercury solution (pH 3) for 24 hours.

Measurement of mercury remediation
After shaking, the mercury solutions were centrifuged (Eppendorf 5810 R) for 15 minutes to separate any remaining peat, vacuum filtered through 0.45 μm nylon filters, then digested. To begin the digestion, the vacuum filtered liquid was transferred to a 300 ml BOD bottle, 5 ml of concentrated sulphuric acid and 2.5 ml of concentrated nitric acid followed by 15 ml of 5 % potassium permanganate solution was added, and the bottle was left to stand for 15 minutes. Next, 8 ml of 5 % potassium persulphate was added and the bottle was placed in a water bath to heat for two hours at 95 °C then cooled to room temperature. If the liquid in the bottle did not appear purple in colour at this stage, more potassium permanganate solution was added until it did. Then, 6 ml of sodium chloride-hydroxylamine sulphate solution was added and the liquid was aerated for 30 minutes to remove any free chlorine, at which stage it became clear and colourless. Any bottle with remaining solids was left overnight to allow the digestion to complete. The mercury concentration was then measured using a Hydra II AA Mercury Analyser (Teledyne Leeman Labs, NH). Before each analysis session, calibration standards (0.2, 0.5, 1.0, 5.0, 10.0, 15.0, and 20.0 ppb) were freshly prepared from the 10 ppm mercury stock solution and used to establish a standard calibration curve.

RESULTS

The results (Table 2) demonstrate that all six peat types worked extremely well at biosorbing mercury from aqueous solutions (92−100 % removal). The pH of the mercury solution had the greatest effect on the mercury removal capacities of the peats, but the effect was slight. Overall, manipulation of the various factors tested did not significantly affect the mercury removal capacities of the peats.

Effect of sample dose
Mixing with a small amount (0.5 g) of all peat types resulted in a substantial removal of mercury from the aqueous solutions. There was no significant change in the percentage of mercury removed as the adsorbent dose was increased from 0.5 g to 2.0 g (99 % for MS, LS and OT peats; 99–98 % for ON peat; 98–99 % for RH peat; 95–99 % for LN peat). The greatest increase (4 %) was for LN peat.

Effect of contact time
For all of the peat samples, increasing contact time from 2 hours to 48 hours did not significantly affect their mercury removal capacities (96 % to 100 % for MS, ON and OT; 97 % to 100 % for LS; 97 % to 96 % for RH; 99 % to 98 % for LN). Four of the six peats (MS, ON, OT, LS) reached their maximum (100 %) mercury removal capacity after a contact time of 48 hours, while the other two peats reached their maximum mercury removal capacity after 2 hours (97 % removal for RH and 99 % removal for LN).

Effect of mixing temperature
There was no significant change in mercury removal among the peat samples as mixing temperature was increased from 24 °C to 40 °C (99 % to 99 % for MS, LS, OT; 99 % to 98 % for ON; 95 % to 96 % for RH; 98 % to 98 % for LN). In other words, temperature did not affect the mercury removal capacity of any of the peats tested.

Effect of mercury solution concentration
Increasing the mercury solution concentration had little to no effect on the mercury removal capacities of the peats. There was no significant change in mercury removal among the peat samples as the mercury solution concentration was increased from 5 ppb to 40 ppb (99 % to 98 % for MS, LS, OT; 98 % to 98 % for ON; 98 % to 95% for RH; 96 % to 96 % for LN).

Effect of pH of mercury solutions
The mercury removal capacities of the peat samples were slightly affected when the pH of the mercury solution was altered. As the pH of the mercury solution increased from 2 to 10, the largest change in percentage of mercury removed occurred with ON peat (99 % at pH 2 to 92 % at pH 10). Mercury removal decreased from 99 % at pH 2 to 95 % at pH

Table 2. Percent mercury biosorption by each of the six peat types with variation of the factors that were manipulated.

factor	value	peat type (sample code)					
		MS	ON	LS	OT	RH	LN
Sample dose (g)	0.5	98.5	98.5	99.0	98.7	97.6	95.3
	1.0	98.3	98.5	98.4	98.6	99.1	97.7
	1.5	98.5	98.0	98.5	98.7	99.0	98.8
Contact time (hr)	2	96.2	96.2	96.9	96.2	97.0	98.8
	24	98.5	98.5	99.0	98.7	95.3	97.6
	48	99.7	99.7	99.7	99.7	96.4	98.4
Mixing temperature (°C)	24	98.5	98.5	99.0	98.7	95.3	97.6
	30	99.0	97.8	98.3	98.4	96.5	98.1
	35	98.2	99.2	98.9	99.0	97.2	98.7
	40	99.0	98.3	98.9	99.0	96.4	98.4
Mercury solution concentration (ppb)	5	98.9	98.4	98.6	98.5	98.3	96.3
	10	97.9	98.0	98.2	98.6	97.4	98.3
	20	98.5	98.5	99.0	98.7	95.3	97.6
	30	98.4	98.6	98.4	98.6	96.5	98.1
	40	97.6	98.0	98.2	98.1	94.9	95.7
pH	2	98.4	99.4	98.8	99.0	95.6	97.1
	3	98.5	98.5	99.0	98.7	95.3	97.6
	4	97.9	92.0	97.9	98.0	95.0	92.7
	5	98.5	94.3	99.7	99.1	95.8	95.2
	6	99.5	96.3	99.0	98.8	96.4	97.0
	7	99.5	95.8	99.1	98.0	96.4	96.9
	8	98.8	94.4	98.0	97.5	95.5	95.3
	9	98.7	93.9	98.7	96.6	95.0	95.4
	10	98.4	92.0	96.1	95.0	95.3	96.2
HCl treatment	without	98.5	98.5	99.0	98.7	97.6	95.3
	with	98.3	98.1	99.4	99.0	94.1	96.7
Re-use	first use	98.5	98.5	99.0	98.7	97.6	95.3
	after desorption	98.3	98.5	98.4	98.6	99.1	97.7
	without desorption	98.5	98.0	98.5	98.7	99.0	98.8

10 with OT peat; and from 99 % at pH 2 to 96 % at pH 10 with LS peat. For the other three peats the change was only 1 % (96 % to 95 % for RH peat, 97 % to 96 % for LN peat) or zero (98 % removal at both pH values using MS peat).

Effect of HCl treatment

Treating the peat samples with HCl had a slight or no impact on their ability to remove mercury from solution. The RH peat experienced the largest change (decrease) in its mercury removal capacity after being treated with HCl (98 % for unaltered sample and 94 % for HCl treated sample). Two of the six peat types experienced a 1 % decrease in their mercury removal capacity after being treated with HCl (99 % for unaltered sample and 98 % after HCl treatment for both MS and ON). The largest increase in mercury removal capacity (95 % for unaltered sample and 97 % for HCl treated sample) was for LN peat. The remaining two peat types (LS and OT) showed no change in mercury removal capacity (99 %) as a result of the HCl treatment.

Desorption of mercury

The desorption results indicate that it may be possible to recover and recycle some of the removed mercury from the peat samples. The MS peat had 41 % of the mercury desorbed, LS and OT had 40 %, LN had 30 %, RH had 26 % and ON had 20 % desorption.

Re-use of samples for biosorption

There was an insignificant difference in mercury bisorption between samples that were re-used after going through desorption and samples being reused without going through desorption. The MS and ON peats had 99 % and the LN peat 97 % mercury removal, whether or not they had been through desorption. The LS and OT peats had 99 % mercury removal when re-used without desorption and 98 % mercury removal when re-used after desorption. RH peat had 96 % mercury removal when re-used without desorption and 97 % mercury removal when re-used after desorption. These results indicate that all six peat types can be re-used for mercury biosorption, whether or not they went through desorption in between the two biosorption cycles.

DISCUSSION

Peat has a unique chemical and physical makeup and possesses a variety and high number of functional groups. The most likely reason that all of the six peat types trialled here worked well at removing mercury from aqueous solutions is the high affinity of their functional groups (Table 1) for mercury ions. These functional groups appear to be capable of removing large amounts of mercury from aqueous solutions.

Manipulating various factors did not significantly affect the mercury removal capacities of the peats. Of the factors whose effects were tested here, the pH of the mercury solution had the greatest (although slight) impact on mercury removal capacities. Both the surface charge of the adsorbent material and the level of ionisation and speciation of the metal are affected by pH (Teker *et al*. 1999). Previous studies have found the optimal pH levels for metal sorption by biomass to be 5−6 and that both highly acidic and highly alkaline environments significantly reduce sorption capacity (Inbaraj & Sulochana 2006, Krishnani *et al*. 2008, Meena *et al*. 2008). At these pH values, there is an increase in available functional groups for metal ion binding as a result of deprotonation. The results of this study indicate that the optimal pH levels for mercury removal are within the range 5−7 for four of the six peat types (pH 5 for LS and OT, pH 6−7 for MS and RH). The ON peat worked best at removing mercury when the pH of the mercury solution was 2, while the LN peat worked best when the pH of the mercury solution was 3. Using three of the peat types (ON, LS and OT), mercury removal decreased when the pH of the mercury solution was ≥ 8. According to Meena *et al.* (2008), this may be due to formation of $Hg(OH)_2$.

Treating the peats with HCl had little or no effect on their ability to remove mercury from solution. It is logical that HCl treatment would not significantly change mercury removal capacity, since the peats in unaltered state removed high percentages of mercury.

All peats were shown to be capable of re-use for mercury biosorption, with or without going through desorption). This means they do not have to be disposed of as a hazardous waste after each mercury biosorption cycle, and they may be repeatedly re-used for this purpose. The desorption results demonstrate that most of the removed mercury can be recovered and recycled from all peat types.

The results of this study demonstrate that all six of the peat types tested have high biosorption potential for mercury in aqueous solution. The materials are economical, environmentally friendly and readily available. Therefore, peat can be effectively used to remove mercury from contaminated water.

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