

## Use of adsorption process to remove organic mercury thimerosal from industrial process wastewater

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### Abstract

Carbon adsorption process is tested for removal of high concentration of organic mercury (thimerosal) from industrial process wastewater, in batch and continuously flow through column systems. The organic mercury concentration in the process wastewater is about 1123 mg/L due to the thimerosal compound. Four commercially available adsorbents are tested for mercury removal and they are: Calgon F-400 granular activated carbon (GAC), CB II GAC, Mersorb GAC and an ion-exchange resin Amberlite GT73. The adsorption capacity of each adsorbent is described by the Freundlich isotherm model at pH 3.0, 9.5 and 11.0 in batch isotherm experiments. Acidic pH was favorable for thimerosal adsorption onto the GACs. Columns-in-series experiments are conducted with 30–180 min empty bed contact times (EBCTs). Mercury breakthrough of 30 mg/L occurred after about 47 h (96 Bed Volume Fed (BVF)) of operation, and 97 h (197 BVF) with 120 min EBCT and 180 min EBCT, respectively. Most of the mercury removal is attributed to the 1st adsorbent column. Increase in contact time by additional adsorbent columns did not lower the effluent mercury concentration below 30 mg/L. However, at a lower influent wastewater pH 3, the mercury effluent concentration decreased to less than 7 mg/L for up to 90 h of column operation (183 BVF).

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### 1. Introduction

Thimerosal is an antimicrobial preservative used in the pharmaceutical and healthcare industry, mainly as a vaccine preservative [1]. It is also used as additive in cosmetics and other biological products to prevent bacterial growth in the cell cultures. The chemical structure of thimerosal is shown in Fig. 1, and Table 1 lists some of its physicochemical properties. Thimerosal contains 46% mercury by weight and is metabolized in the human body to thiosalicylic acid and ethyl mercury (mainly excreted in the feces as inorganic mercury) [2]. Thimerosal is likely to be present in waste streams from hospital, clinical laboratories and pharmaceutical industries. At present, there is very little information on the specific remediation techniques for the treatment of wastewater containing thimerosal.

It is necessary to further investigate and identify efficient treatment methods for removal of thimerosal from wastewaters. Fortunato et al. [3] examined biological degradation of thimerosal. They used *P. putida spi3* to remediate thimerosal contaminated vaccine wastewater. The thimerosal concentration in the wastewater was about 50 mg/L as thimerosal and about 24 mg/L as mercury. The bacteria transformed thimerosal to Hg<sup>0</sup>, which would remain in aqueous, sludge or be volatilized to the gaseous phase. The process required specific conditions for the mercury resistant *pseudomonas putida* strain to grow.

Among other reported techniques for the treatment of wastewater containing organic mercury, adsorption process shows good potential and can be cost efficient. Logsdon et al. [4] reported methyl mercury and inorganic mercury removal using both granular activated carbon (GAC) and powdered activated carbon (PAC). For their column studies with GAC, average influent concentration ranged between 20 and 29 µg/L. We have previously investigated the removal of thimerosal from pharmaceutical wastewater using GAC columns at bench and pilot scale [5,6]. These tests were performed at low mercury concentrations

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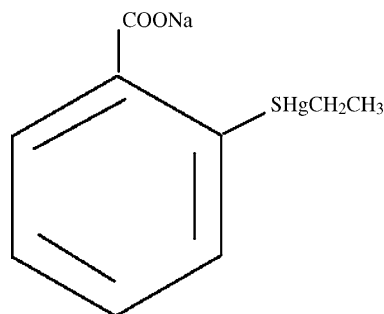


Fig. 1. Chemical structure of thimerosal.

Table 1  
Select properties of thimerosal

Molecular formula	C <sub>9</sub> H <sub>9</sub> HgNaO <sub>2</sub> S
Molecular weight	404.8
Density <sup>a</sup>	0.93 g/cm <sup>3</sup>
Molar volume <sup>a</sup>	435 cm <sup>3</sup> /g mol
Appearance	Light cream-colored crystalline powder; light sensitive
Melting point <sup>a</sup>	232–233 °C
Water solubility <sup>a</sup>	≥10 g/100 mL at 19 °C
pK <sub>a</sub> <sup>b</sup>	6.7

<sup>a</sup> Obtained from Chemfinder database.<sup>b</sup> Ref. [15].

of about 4 mg/L. It was observed that the carbon adsorption process was effective for 99.8% removal of thimerosal.

However, industrial process wastewater may contain high concentrations of thimerosal. There is no data reported in the literature on removal of high thimerosal concentrations from wastewater, in excess of 1000 mg/L. The adsorption process could be effective for treatment of high thimerosal concentrations wastewater. In addition, adsorption conditions such as wastewater pH and empty bed contact time (EBCT) could have significant impact on the removal effectiveness.

This paper examines the use of adsorption process for removal of high concentrations of thimerosal (1000 mg/L as Hg) from industrial process wastewater in both, batch and continuously flowing column systems. Effects of pH, EBCT and the type of adsorbents were evaluated. The adsorbents tested were: F-400 GAC, Mersorb GAC, CB II GAC and Amberlite GT73 resin. Removal of color, turbidity and change in pH of the process wastewater was also examined.

Table 2  
Select adsorbent properties as reported by the manufacturer

Adsorbent	Type	Appearance	Surface area <sup>a</sup> (m <sup>2</sup> /g)	Bulk density (g/L)	Impregnation
F-400	Activated carbon	Black granules	1050	480	None
CB II	Activated carbon	Black granules	1150	480	Sulfur (18 wt%)
Mersorb	Activated carbon	Black cylindrical granules	1000	550	Sulfur
GT73	Ion-exchange resin	Moist spherical yellow beads	55	800	Thiol and minor sulfonic acid groups

<sup>a</sup> N<sub>2</sub> BET area.

## 2. Experimental

### 2.1. Materials

F-400 GAC was purchased from Calgon Carbon Corporation, Huntington, WV; CB II was purchased from Barnebey & Sutcliffe Corporation, Columbus OH; Mersorb was purchased from Nucon International Inc., Columbus, Ohio; and Amberlite GT73 was purchased from Supelco, Bellefonte, PA. Both Mersorb and CBII are sulfur impregnated GACs. Table 2 lists select properties of these adsorbents. ACS plus grade nitric acid, sulfuric acid, potassium permanganate, potassium persulfate and hydroxylamine hydrochloride reagents were obtained from Fisher Scientific. Thimerosal was obtained from Fisher Scientific. Stannous chloride was obtained from Sigma–Aldrich. Nitric acid and NaOH solution were used for pH adjustment. Nitric acid was also used for glassware washing. The process wastewater used in the study contained organic mercury thimerosal, with an approximate concentration of 1123 mg/L as mercury. The pH ranged from 8.8 to 9.5, and the solution had a predominant pink color. The suspended solids concentration was approximately 37 mg/L. The process wastewater was from production of flue vaccine and had high dissolved solids concentration of about 1,712 mg/L.

### 2.2. Procedure and equipment

Batch isotherm tests were performed for the initial screening of the adsorbents. Bottles containing known amount of adsorbent were filled with process wastewater at certain solution pH (3.0, 9.5 or 11.0). The bottles were shaken periodically for six days at 25 ± 2 °C. Aqueous samples from the bottles were analyzed for mercury concentration for a six day period. Data from previous isotherm study with thimerosal and GAC showed that six days were sufficient for the adsorption equilibrium to occur [5]. Control experiments, without the addition of adsorbent, were performed at each pH simultaneously with the adsorption experiments. The concentration of mercury adsorbed on the adsorbent was calculated from the difference between the initial concentration and final equilibrium concentration in the solution.

Column studies were performed with four to six columns-in-series. Glass columns of 5 cm diameter and 30 cm length, fitted with Teflon end caps were connected in series, as shown in Fig. 2. The process wastewater was pumped from the influent reservoir tank at a rate of 17 mL/min through the column system. The GAC adsorbents were soaked for one day in deionized water, before

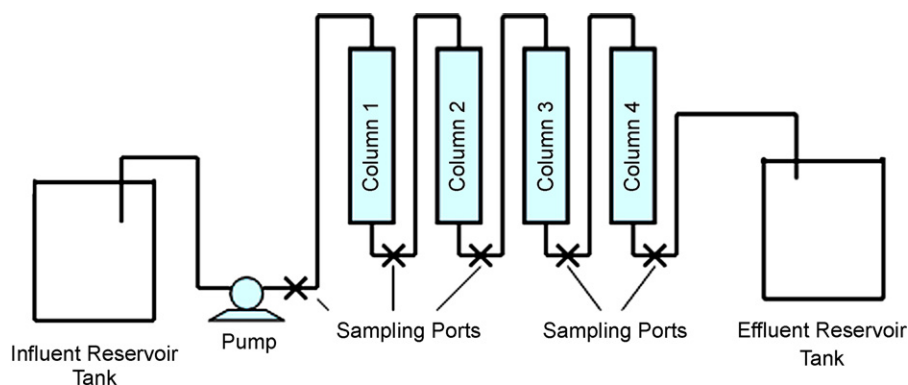


Fig. 2. Schematic of the experimental set-up for the four-column-in-series study.

being added to the columns. Each column contained about 240 g of carbon and was designed for 30 min EBCT. The influent and effluent from each column during the experiment were sampled and analyzed for total Hg concentration. Certain samples were also analyzed for pH, color and turbidity.

### 2.3. Analytical methods

All glassware was acid washed with dilute nitric acid and rinsed with deionized water before use. Mercury was analyzed using EPA Cold Vapor Atomic Absorption Method 245.1 [7]. The mercury analyzer was a Buck Scientific, Model 400A with sensitivity equal to 0.01  $\mu\text{g}$  of mercury, range of scale from 0–9  $\mu\text{g}$  and a response time of less than 0.5 min. The absorption cell consisted of a quartz window of 125 mm path length. The sample (100 mL) was treated with 2.5 mL of concentrated nitric acid, 5 mL of sulfuric acid in presence of 15 mL of potassium permanganate and 8 mL of potassium persulfate, in a hot water bath for 2 h at 90 °C to digest organic mercury to mercuric (Hg(II)) form. After cooling back to room temperature, 6 mL of hydroxylamine hydrochloride was added to reduce the excess permanganate. Lastly, 5 mL of stannous chloride was added to reduce the mercury, and the bottle was immediately attached to the aeration apparatus to air strip the mercury into the absorption cell of the spectrophotometer.

Standard solutions were prepared with thimerosal in milli-Q water to obtain 100 mL samples containing 0.5, 2, 4, and 6  $\mu\text{g}$  of mercury. Standards were analyzed in duplicate and the data showed excellent reproducibility (R.S.D. < 6%). The detection limit of the method was 1  $\mu\text{g/L}$ . All concentrations reported are for total mercury.

Color, pH and turbidity were measured using a Hach DR-A colorimeter, an Oakton pH meter and a Hach 2100N turbidimeter, respectively.

The mass of mercury removed in the column was calculated by multiplying the flow rate (L/h) with the integrated area (mg h/L) between the influent and effluent profiles from the concentration-time plots (breakthrough figures). The mercury loading on the adsorbent in the column was the ratio between the mass of mercury removed and the mass of GAC in the column.

## 3. Results and discussion

The characteristics of the process wastewater used in this study are shown in Table 3. Total mercury present was due to organic mercury and averaged about 1123 mg/L. No inorganic mercury was observed (as measured without the digestion step which is described in Section 2.3).

### 3.1. Batch experiments

In this study, three types of GACs (F-400, Mersorb and CB II) were used. They all have surface areas higher than 1000  $\text{m}^2/\text{g}$ , as shown in Table 2. High surface area would be beneficial for higher adsorption of organic mercury. Mersorb and CB II GACs were used to observe the effect of impregnated sulfur as some studies have reported that impregnated sulfur provides enhanced mercury adsorption [8,9]. Sulfur has a high affinity for Hg ( $\log K_{\text{sp}} = -52.7$ ) [10]. Certain ion-exchange resins such as GT73 have been reported to be effective for mercury removal. GT73 carries thiol and sulfonic acid functional groups which could enhance mercury chemisorption. The Amberlite GT73 ion-exchange resin was evaluated and compared with selected GACs for mercury adsorption.

Fig. 3 shows thimerosal adsorption versus adsorbent dosage with different adsorbents at three different solution pHs. The thimerosal concentration in the wastewater was the same for these tests. Upon lowering the solution pH to 3, some precipitate was observed in the wastewater. The precipitate was yellow–brown in color and was not identified. The Hg

Table 3  
Characteristics of the process wastewater (as received)

pH	8.8–9.5
Suspended solid (mg/L)	37
Dissolved solid (mg/L)	1712
Average total mercury (mg/L)	1100–1300
Ionic mercury Hg(II) (mg/L)	ND
Average organic mercury (mg/L)	1100–1300
Color (CU)	400
Turbidity (NTU)	312

ND: not detected; CU: color unit.

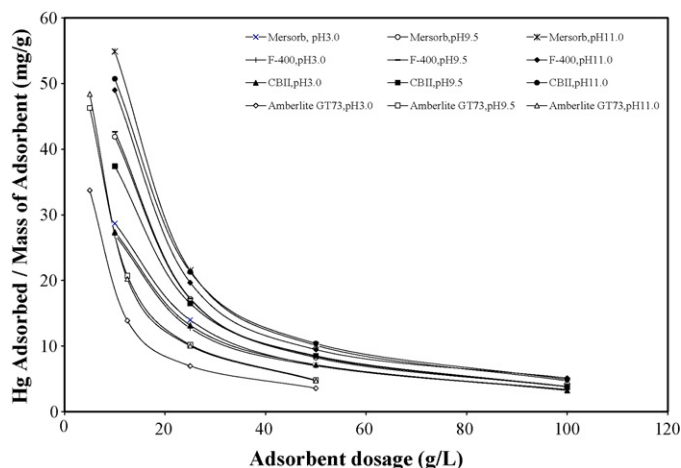


Fig. 3. Thimerosal adsorption with different adsorbents and pH, in batch experiments; ambient temperature (25 °C).

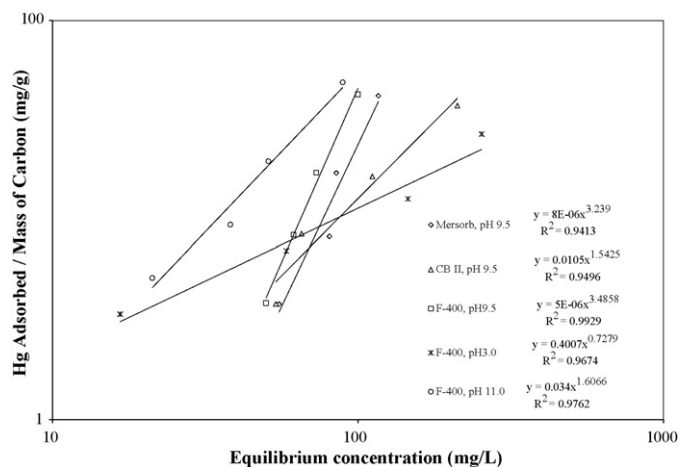


Fig. 4. Adsorption isotherms for F-400 GAC at pH 3.0, 9.5 and 11.0; for CB II GAC at pH 9.5; and Mersorb GAC at pH 9.5; ambient temperature (25 °C).

concentration in the supernatant after precipitation reduced to about 700 mg/L, which has been accounted in calculating the adsorption effects (i.e., 700 mg/L was used as the initial concentration for calculating the adsorbent loading). It may be observed in Fig. 3 that higher adsorbent dosage resulted in lower thimerosal adsorption loading (mg Hg/g adsorbent). All three GACs showed similar adsorption loading. Amberlite GT73 showed low adsorption loading, which could be attributed to its lower surface area.

Table 4  
Freundlich isotherm parameters of three GAC adsorbents for mercury wastewater under different pH; room temperature

Adsorbent	Freundlich exponent, $1/n$ ( $r^2$ )			Freundlich adsorption constant ( $K_f$ )		
	pH 3.0	pH 9.5	pH 11.0	pH 3.0	pH 9.5	pH 11.0
Mersorb	1.11 (0.85)	3.24 (0.94)	2.06 (0.65)	0.0842	0.00001	0.00370
CB-II	0.78 (0.95)	1.54 (0.95)	1.43 (0.97)	0.3631	0.01050	0.05330
F-400	0.73 (0.97)	3.49 (0.99)	1.61 (0.98)	0.4007	0.00001	0.03400

$n$  = dimensionless;  $K_f$  is in (L/mg)<sup>1/n</sup> (mg/g).

Fig. 4 shows the adsorption loading (mg Hg/g adsorbent) versus equilibrium adsorbate concentration (mg Hg/L) for F-400 GAC at pH 3.0, 9.5 and 11.0; CB II GAC at pH 9.5; and Mersorb GAC at pH 9.5. The adsorption of thimerosal on GACs at pH 3.0, 9.5 and 11.0 was described by the Freundlich isotherm equation (Eq. (1)). In Eq. (1),  $C_s$  represents the mercury adsorbed on the carbon;  $C_e$  is the equilibrium mercury concentration in the solution;  $K_f$  represents the Freundlich adsorption constant; and  $1/n$  is the Freundlich exponent. The related  $K_f$  and  $1/n$  values for each carbon adsorbent under different pH are listed in Table 4. Adsorption at low mercury concentrations (<10 mg/L) using Freundlich model has been previously reported for ionic mercury and thimerosal [5].

$$C_s = K_f C_e^{1/n} \quad (1)$$

Solution pH can influence the adsorption process. It has been reported [5] that at low mercury concentration (<10 mg/L), pH 7 or lower was favorable for adsorption of ionic mercury, while pH did not have a significant influence on adsorption of thimerosal on GAC. However, in this study at high thimerosal concentration, pH has a significant influence on adsorption (Table 4). For the three GACs, Freundlich exponent ( $1/n$ ) was lowest at pH 3, as shown in Table 4. Freundlich adsorption constant  $K_f$ , which represents the adsorption capacity, was the highest at pH 3. This means that acidic conditions are favorable for thimerosal adsorption. The pH can affect the chemical form of thimerosal, functional groups on carbon surface and the interaction of background chemicals in the process wastewater with carbon. Many commercially available activated carbons have a point of zero charge (PZC) above pH 7.5 [11,12]. For example, the PZC of F-400 GAC was reported to be 7.9 [13] or 8.5 [14]. Therefore, the carbon surface is negatively charged at pH higher than 7.5. The  $pK_a$  of thimerosal is 6.7 [15]. At higher solution pH thimerosal will be predominantly present in ionic form, and which would not be a favorable condition for its adsorption on to the negatively charged carbon surface.

### 3.2. Column experiments

Considering the thimerosal adsorption loading observed in batch experiments (Fig. 4), GAC columns of 30, 60, 90 and 120 min EBCT were designed and tested. The effect of solution pH, GAC type and EBCT to achieve low effluent mercury concentration was examined. Four columns-in-series studies were conducted and are described below. Table 5 summarizes the operational parameters of each F-400 GAC column.



Table 5

Operational parameters for each F-400 GAC column-in-series experiments in study A, B, C and D

Operational parameter	Values
EBCT (min)	30
Flow rate (mL/min)	17
Superficial loading velocity (cm/min)	0.87
Column diameter (cm)	5
Bed depth (cm)	25.6
Adsorbent mass (g)	240
Bulk density (g/mL)	0.48 (F-400 and CBII GAC)

### 3.2.1. Study A: 120 min EBCT F-400 GAC; pH 9.5

Four F-400 GAC columns-in-series each of 30 min EBCT were used. The total EBCT of the system was 120 min (a total of 960 g of carbon). The process wastewater at pH 9.5 was passed through the four columns at 17 mL/min for a 79 h test period. The average influent mercury concentration was 1123 mg/L. The adsorption results from column study A are shown in Fig. 5. The mercury concentration in the effluent decreased after each F-400 GAC column. For analysis purposes, Hg concentration of 30 mg/L was considered as the breakthrough point for each column, which represented about 97% of thimerosal removed. The entire system could operate for approximately 47 h before the breakthrough from the last column occurred (Fig. 5), treating about 48 L of wastewater (96 Bed Volume Fed (BVF)). Fig. 6 shows the total mercury removed in each column for the 47 h period at breakthrough. About 63% of the mercury removal occurred in the 1st column, and 23%, 11% and 3% in columns 2, 3 and 4, respectively. F-400 GAC in the 1st column removed more thimerosal than the rest of the columns, and the mass of mercury removed decreased from column 1 to 4. This is expected since the column receiving higher influent concentration would contribute more to the removal of mercury. Similar observations were made in a previous study [6], where under low initial mer-

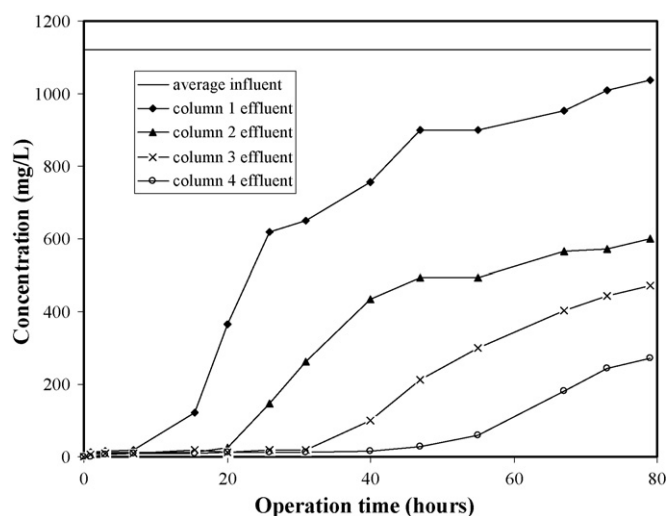


Fig. 5. Mercury breakthrough data from column study A: four-columns-in-series; 120 min EBCT F-400 GAC; pH 9.5; average influent concentration: 1123 mg/L.

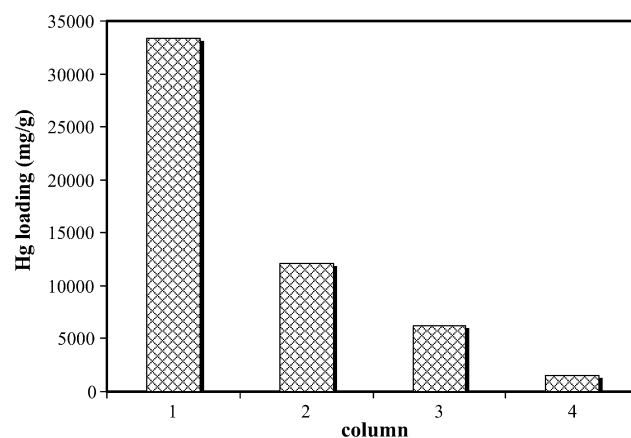


Fig. 6. Mercury removed by each column at the breakthrough in study A.

cury concentration ( $\sim 4$  mg/L), almost 99.5% of mercury was removed by the 1st column while only 0.3% was removed by the 2nd column at breakthrough.

It may be observed that the column 1 operated for 7 h to produce an effluent concentration lower than 30 mg/L. This represents 0.03 L of wastewater treated per gram of GAC (7 L treated by 240 g GAC in column 1) or 34 g GAC used per liter of process wastewater. From Fig. 3, if 34 g of carbon is used for one liter of process wastewater in the batch system, the equilibrium aqueous concentration would be approximately 600 mg/L. In a column, the adsorbent upon saturation is in equilibrium with the influent concentration. In the batch system, the adsorbent is in equilibrium with the effluent concentration. Hence, the adsorbent loading would be lower in the batch system. Moreover, in columns-in-series approach it is not necessary to change the carbon in the entire system (i.e. all the columns) when the breakthrough occurs. As a cost effective method, only the carbon from the 1st column would need to be replaced, and the 2nd column could be used as the 1st column.

The change in pH and removal of color and turbidity were also examined during the study. No significant difference in influent and effluent pH was observed. The color breakthrough occurred immediately, and an average of 28% color removal occurred in the system. The turbidity breakthrough occurred almost immediately, and no significant reduction in turbidity was observed (results not shown here).

### 3.2.2. Study B: 180 min EBCT F-400 GAC; pH 8.8

To examine the possibility of using higher EBCT to further decrease the effluent concentration of thimerosal, two additional GAC columns were added. The EBCT of the system increased from 120 to 180 min (a total of 1440 g of carbon). The average initial mercury concentration of the process wastewater used for this study was 1306 mg/L, and the pH was 8.8 (as received). The mercury breakthrough profiles from this study are shown in Fig. 7. With the six columns-in-series system, the effluent concentration of mercury was below 30 mg/L for 97 h of operation. This represents about 99 L of wastewater treated (197 BVF). With the increase in the amount of carbon, the system operated for 50 more hours as compared to Study A (four columns

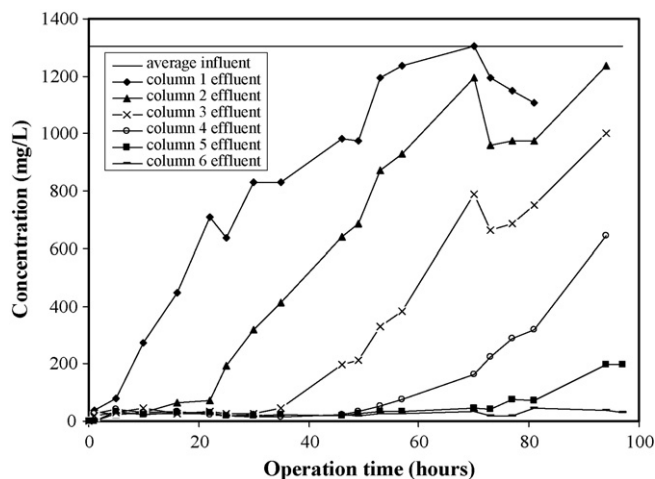


Fig. 7. Mercury breakthrough data from column study B: six-columns-in-series; 180 min EBCT F-400 GAC; pH 8.8; average influent concentration: 1306 mg/L.

of 120 min total EBCT) before breakthrough of 30 mg/L was observed. It seems that there was a threshold concentration ranging from 20 mg/L to 30 mg/L of mercury in the effluent with 180 min EBCT F-400 GAC columns. This may be due to the interference of background organics on thimerosal adsorption. Study by Mohan et al. [16] also suggested that presence of background chemicals could influence and compete with mercury adsorption on the carbon columns.

### 3.2.3. Study C: 90 min EBCT F-400 GAC + 30 min EBCT Mersorb + 30 min EBCT CB II (total 150 min EBCT); pH 9.5

Two sulfur impregnated adsorbents (Mersorb and CB II GAC) were also evaluated to examine the feasibility of achieving lower mercury concentration in the effluent. Sulfur impregnation can provide higher removal of mercury due to its high affinity for mercury [8]. In this study, three F-400 GAC columns, one Mersorb GAC column and one CB II GAC column were used in a five columns-in-series system. Each column was of 30 min EBCT and the column study was operated for 35 h. Mercury breakthrough results are shown in Fig. 8. The concentration of mercury was reduced from an average of 1196 mg/L in the influent to below 30 mg/L in the effluent during the 35 h operation period. However, Mersorb and CB II GAC columns did not show a significant enhancement in mercury removal as compared to F-400 GAC in studies A and B.

### 3.2.4. Study D: 120 min EBCT F-400 GAC; pH 3

The isotherm study in Fig. 4 showed that pH 3 was favorable for adsorption of thimerosal. Hence, the pH of the process wastewater was adjusted to 3 with nitric acid to examine the impact of lower pH on thimerosal adsorption in column system. Four F-400 GAC columns, each of 30 min EBCT were used in-series. Upon lowering the solution pH to 3, the Hg concentration in the influent tank reduced to about 700 mg/L. The supernatant was passed through the four F-400 GAC columns-in-series system and the effluent data is presented in Fig. 9. The mercury concentration decreased significantly from the average influent

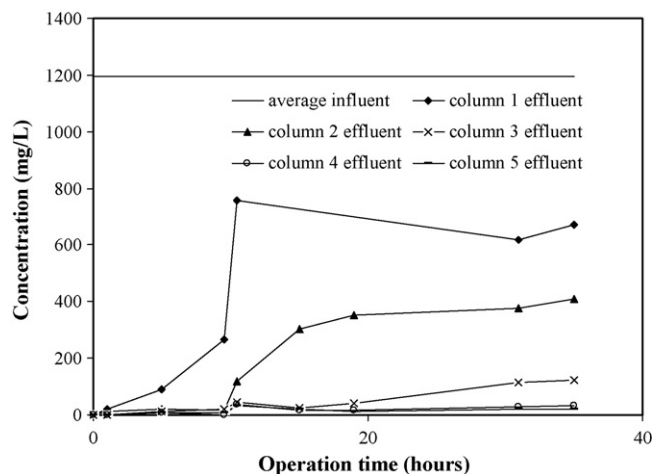


Fig. 8. Mercury breakthrough data from column study C: five-columns-in-series; total 150 min EBCT; pH 9.5; average influent concentration: 1196 mg/L.

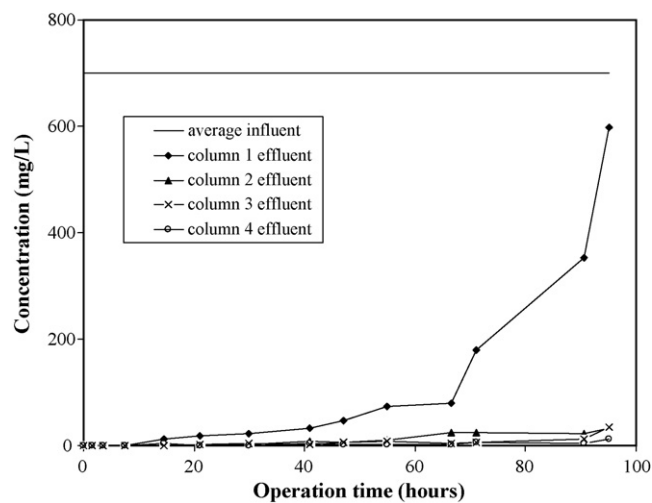


Fig. 9. Mercury breakthrough data from column study D: four-columns-in-series; 120 min EBCT F-400 GAC; pH 3; average influent concentration: 700 mg/L.

of 700 mg/L to less than 7 mg/L in the effluent for an operation time of 90 h. This corresponds to 92 L of wastewater treated and 183 BVF. It is likely that due to the removal of some background material by precipitation, there was less interference of background material during thimerosal adsorption. Lower pH was favorable to achieve lower effluent concentration of mercury. The batch isotherm study also showed favorable adsorption at pH 3 for F-400 GAC (Fig. 4). The first column removed about 88% of the mercury during the 90 h of operation. The mercury loading on the GAC in column 1 was 119 mg/g at breakthrough concentration of 30 mg/L for 41 h of operation.

## 4. Conclusion

The batch isotherm tests showed that the Freundlich adsorption model can describe thimerosal adsorption for F-400, CB II and Mersorb GACs at solution pH 3, 9.5 and 11. Amberlite GT73 ion-exchange resin had lower thimerosal adsorption capacity as compared to the GACs. Solution pH has a significant influence

on thimerosal adsorption. Freundlich exponent ( $1/n$ ) was lowest at pH 3 and Freundlich adsorption constant  $K_f$  was highest at pH 3. Hence, acidic pH was favorable for thimerosal adsorption on to the three GACs examined in this study.

The GAC column system removed more than 97% of the thimerosal present in the process wastewater. Four F-400 GAC columns-in-series (120 min total EBCT) showed that process wastewater with an initial concentration of 1123 mg/L and pH 9.5 (as received) could be treated to produce an effluent mercury concentration of 30 mg/L for 47 h of operation (96 BVF). This represents treatment of 48 L of process wastewater with about 1 kg of GAC. There is a threshold effluent mercury concentration ranging from 20 to 30 mg/L. Usage of more F-400, CB II or Mersorb GACs did not show a significant decrease in effluent mercury concentration. At solution pH 3, four GAC columns-in-series with 120 min total EBCT operated for up to 90 h (183 BVF). This represents treatment of 92 L of process wastewater with an average influent thimerosal concentration of 700 mg/L mercury. The effluent mercury concentration was less than 7 mg/L, with a weighted average effluent concentration of 3.2 mg/L.

Lowering the solution pH to 3 was beneficial in lowering the effluent mercury concentration and extending the column system operation time. A precipitate was observed to occur in the influent wastewater at pH 3. The mercury concentration in the influent was lower at pH 3 due to the precipitation. The increased cost of acidification before treatment, pH neutralization after treatment, clarifier or filtration and disposal of sludge has to be considered as this would involve additional cost. If lower mercury concentration in the effluent is desired, additional pre and post treatment will have to be evaluated. These methods may include chemical precipitation as pre-treatment or membrane separation as post-treatment.

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