



Stabilization/solidification (S/S) of mercury-containing wastes using reactivated carbon and Portland cement

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Received 11 September 2001; received in revised form 16 January 2002; accepted 17 January 2002

Abstract

Stabilization/solidification (S/S) of mercury-containing solid wastes using activated carbon and cement was investigated in this study. The activated carbon used in the study was a powder reactivated carbon (PAC). The effect of sulfur-treatment of the PAC was also studied. It was found that PAC was effective in stabilizing Hg in the waste surrogate. Pretreatment of the PAC by soaking it in CS₂ significantly improved the mercury adsorption capacity of the PAC. The adsorption equilibrium was reached within 24 h. The optimum pH for the reaction was within the range of 5.0–5.5. After mercury stabilization by adsorption on the reactivated carbon, the Hg waste surrogate was mixed with Portland cement for solidification. Surrogates with up to 1000 mg/kg Hg were stabilized and solidified well enough to pass the TCLP test. The adsorption of mercury by reactivated carbon was in accordance with the Freundlich isotherm. Cement solidification of reactivated carbon-stabilized surrogates, significantly reduced the often-reported interference by chloride ions, by forming a barrier outside of the carbon particles. The S/S process using reactivated carbon and cement is an effective and economical technology for treating and disposing mercury-containing solid wastes. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Mercury; Activated carbon; Stabilization; Solidification; Sulfur

1. Introduction

The toxic nature of mercury and its compounds has been known for centuries. Mercury persists in the environment and often creates long-term contamination problems. It is possible for inorganic mercury to be biologically methylated. Methyl mercury has high affinities for fatty tissues in organisms and can accumulate to toxic levels within those

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organisms. Food chain transport and bioaccumulation of mercury have been well documented. Therefore, strict control of mercury leaching from mercury-containing wastes is required. Mercury-containing solid wastes are generated by a wide variety of industries such as chloralkali, paint, pulp and paper, oil refining, rubber processing and fertilizer [1]. Heavy metals, such as Pb, Cd and Cr, have been successfully immobilized using stabilization/solidification (S/S) technologies. The conventional S/S technologies, however, cannot effectively reduce the leachability of mercury [2].

Many researchers have studied the use of activated carbon for the adsorption of mercury species from the gas phase [1,3–7]. The effect of sulfur-impregnation of the activated carbon surface has also been investigated [8–11]. Activated carbons have been proven to be effective in removing both mercury(II) and elemental mercury from aqueous solutions and vapor phases. Reaction mechanisms involved in mercury capture by activated carbon are complex. Chemisorption and physisorption could all be very important. After sulfur-impregnation, the mercury adsorption capacity of activated carbons has been greatly enhanced. This is possibly due to the formation of mercuric sulfide on the carbon surface [8]. Huggins et al. [3] used XAFS spectroscopy to examine mercury sorption on activated carbon. They found that the activating element (S) formed a sorption complex with mercury on the surface of the sulfur-activated carbon. SEM micrographs of the fresh carbon and the carbon after mercury adsorption showed that mercury is adsorbed on the surface on particular sites where high sulfur concentrations exist [4]. On the other hand, Vitolo and Pini [12] found that further sulfur deposition on a commercial pre-sulfurized activated carbon drastically reduced the carbon's mercury chemisorbing capacity. Valenzuela-Calahorra et al. [13] studied several methods to introduce sulfur into activated carbon. They found that the adsorption of mercury(II) from aqueous solution is highly dependent on the method followed for introducing sulfur into the activated carbon. Their study also showed that the carbon-sulfur complex is highly stable.

In a study of heavy metals removal by Fe-oxide impregnated activated carbon (FeAC), Reed et al. [14] found that, for the cationic Hg, removal by the FeAC was slightly higher than with the non-impregnated carbon. It could be assumed that heavy metals on the carbon surface might not work as catalysts for mercury adsorption.

So far, there has been little application of activated carbon in mercury-containing solid waste S/S processes, due to the high cost of activated carbon. The major objective of this study was to find a cost-effective pathway to treat and dispose the mercury-containing solid wastes. In this study, a low-cost powder reactivated carbon (PAC) was used to stabilize mercury in solid wastes. The reactivated carbon can be obtained at only a fraction of the cost of virgin PAC. In addition, the PAC was treated with CS₂ to enhance its adsorption capacity, according to the literature mentioned above. The stabilized solid wastes were then subjected to cement solidification to test the effectiveness of the whole S/S process.

2. Materials and methods

2.1. Regenerated powder activated carbon (PAC)

A PAC was used in this study. Calgon Carbon Company thermally reactivated the carbon from activated carbons that had previously been used in various applications. The BET

Table 1
Characteristics of test sand

Source	Cube test sand (CT-109A ELE International)
Specific gravity	2.65
Bulk density	93.45 lb/ft ³ (1.497 g/cm ³)
Grading	Sieve No. 30 retains 2% Sieve No. 40 retains 30% Sieve No. 50 retains 75% Sieve No. 100 retains 98%

surface area of the carbon is $966 \pm 50 \text{ m}^2/\text{g}$. It is relatively inexpensive, with the cost less than one-fourth that of virgin activated carbon. To improve the adsorption capacity of the PAC, it was treated with CS_2 . For CS_2 treatment, the PAC was soaked in aqueous CS_2 and mixed for 48 h, and then the mixture was dried in a hood on a heat-stir plate (Thermolyne Cimarec 2) with minimum heating. CS_2 treated PACs are labeled as PAC-s in all figures in this paper, while conventional PAC is labeled as PAC.

2.2. Surrogate wastes

Mercuric nitrate solutions were used in the kinetic and pH effect studies. The solutions used in these studies contained 40 mg/l Hg^{2+} and were prepared using $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ and DI water.

Pure cube test sand (CT-109A ELE International) was used as the solid waste surrogate, so as to minimize competitive adsorption by substances in real-world soils (Table 1). The surrogates were made by mixing together $\text{Hg}(\text{NO}_3)_2$, pure cube test sand and 4% DI water. Then, the surrogates were put in a hood for drying and aging for 2 weeks. The total Hg concentrations of the surrogates were 100, 250, 500, 750, and 1000 mg/kg. A 10:1 DI water/surrogate ratio was used for all surrogate experiments.

2.3. Mercury speciation

MINTEQA2 is a geochemical equilibrium speciation computer model distributed by the US EPA [15]. The model is capable of calculating equilibrium aqueous speciation. In the study, the model was used to calculate mercury speciation of HgS and $\text{Hg}(\text{NO}_3)_2$ in order to further understand the behavior of mercury.

2.4. Batch adsorption studies

2.4.1. Adsorption at various pH values

The study of PAC adsorption at various pH values was carried out over the pH range of 2.0–12.0. Fifty millilitre of mercuric nitrate solution containing 40 mg/l Hg^{2+} , and 10 g of surrogate containing 1000 mg/kg Hg^{2+} were used in the pH experiments. Nalgene HDPE bottles (125 ml) were used in the experiments. For studies using the mercuric nitrate solutions, 1 g PAC or PAC-s was added into each bottle. For solid waste surrogates, first 100 ml of DI water and then 1 g PAC or PAC-s were added into each bottle. All bottles were shaken

for 24 h. The pH was manually maintained constant by adding 1.0 or 0.1N HNO₃ or NaOH solutions. After completion of the experiments, the mixtures were filtered through 0.45 μm filters, and the filtrates were analyzed for mercury.

2.4.2. Batch kinetic studies

Batch kinetic studies were carried out on both Hg(NO₃)₂ solutions and solid waste surrogates to determine the effect of contact time on adsorption. Fifty millilitre of mercuric nitrate solution containing 40 mg/l Hg²⁺ was transferred to 125 ml plastic bottles. For the surrogates, 10 g of surrogate containing 500 mg/kg Hg²⁺ and 100 ml DI water were transferred to 125 ml plastic bottles. Then, 0.1 and 0.2 g PAC-s were introduced into each bottle for mercuric nitrate solution and surrogate, respectively. The reaction mixture was shaken at room temperature. The mixture pH was manually maintained at 5.5 by adding 0.1N HNO₃ or NaOH solutions. At preset time periods, i.e. 10, 20, 30 min, 1, 2, 4, 8, 24, 48, 72, 96, and 120 h, samples were taken and filtered through 0.45 μm filters. A control sample with no PAC or PAC-s was also run to check if the plastic bottle adsorbed any mercury during the experiment. The equilibrium time for adsorption of mercury by PAC-s was determined by analyzing the filtrates for mercury.

2.4.3. Interferences

Possible interferences with Hg immobilization by other anions and cations present in solution were investigated. The anions and cation investigated were: Cl⁻, PO₄³⁻, Ac⁻, SO₄²⁻, CO₃²⁻, and Pb²⁺. PAC-s, in the amount of 0.5 g, was added to 10 g of surrogate with various Hg concentrations. Then, 100 ml of certain interference solutions were added into the mixture. A series of control samples was run by adding 100 ml of DI water into the mixture. The reaction mixtures were shaken for 24 h. The mixture pH was manually maintained constant at 5.0 by adding 1.0 or 0.1N HNO₃ or NaOH solutions. After 24 h, the mixtures were filtered through 0.45 μm filters, and the filtrates were analyzed for mercury. The solid phase was dried at 60 °C in an oven. Part of the dried solids was subjected to the TCLP leaching test. The rest of the dried solids was solidified with Portland cement, then crushed and subjected to the TCLP leaching test.

2.5. Ordinary Portland cement (OPC) solidification

Type I ordinary Portland cement (OPC) was used in solidification experiments. After reactivated carbon stabilization, the solid waste surrogate was mixed with OPC for solidification. The surrogate/OPC ratio used was 1:1. The water/OPC ratio used was 0.5. After setting for 5 days, the cement paste mixture was crushed and subjected to the TCLP test. The particle size of the crushed samples less than 9.5 mm, according to the requirement of the TCLP procedure. A series of control samples was produced by mixing OPC and un-stabilized surrogates, using the ratios mentioned above.

2.6. Analytical methods

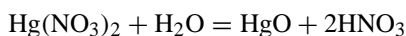
A Perkin-Elmer Analyst 300 atomic absorption spectrophotometer equipped with a FIAS 100 cold vapor analyzer was used to analyze total Hg²⁺ concentrations. All samples were

prepared with standard liquid and solid Hg digestion methods [16]. Solution pH was measured with an Accumet AR50 pH meter. The balance used was a Mettler Toledo B303. A Fisher Scientific Isotemp Oven (Model 615G) was used to dry solid samples. A New Brunswick Scientific reciprocal water bath shaker was used to shake adsorption samples. All experimental treatments in this study were conducted with 10% of the samples repeated in triplicate. All experiments used acid-washed (50% HNO₃) HDPE plastic bottles. TCLP leaching tests were carried out in an end-over-end 30 ± 2 rpm tumbler. All experiments were carried out using MilliQ water. The mercury standard was purchased from Fisher. All other chemicals were of analytical standard quality.

3. Results and discussion

3.1. Mercury speciation

Fig. 1 shows the computed mercury speciation in a mercuric nitrate solution containing 40 mg/l Hg²⁺ using MINTEQA2. It should be pointed out that mercuric nitrate can be completely decomposed into HgO and nitric acid by a large excess of water [17].



The solubility of HgO is about 51 mg/l at 25 °C. Therefore, mercuric nitrate is completely dissolved in water for a concentration of 40 mg/l Hg²⁺, over the pH range of 2–12. Major species of concern in the pH range 2–12 are Hg²⁺, HgOH⁺, and Hg(OH)₂⁰. At pH = 2, the dominant species is Hg²⁺. Hg(OH)₂⁰ gradually becomes the dominant species from pH 2 to 4. At pH = 4, over 95% of the aqueous Hg(II) exists as the neutral Hg(OH)₂⁰.

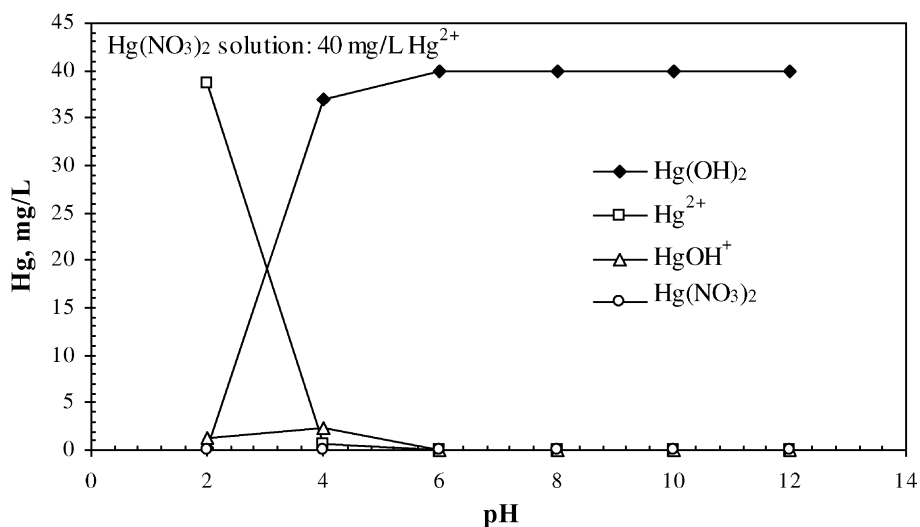


Fig. 1. Mercury speciation in a mercuric nitrate solution by MINTEQA2.

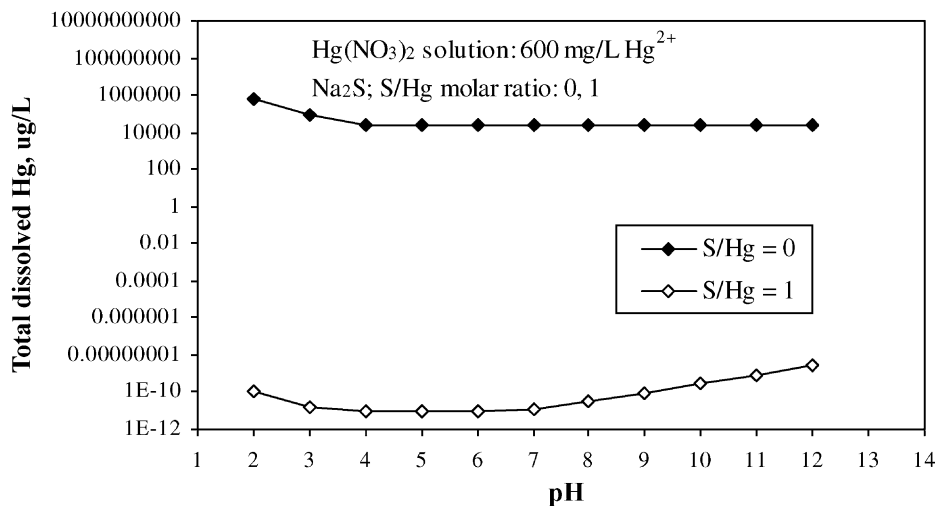


Fig. 2. HgS solubility simulation by MINTEQA2.

HgS solubility was also simulated using MINTEQA2 (Fig. 2). During simulation, certain amounts of Na₂S were added into the Hg(NO₃)₂ solution, with a concentration of 600 mg/l Hg²⁺, to achieve various S/Hg molar ratios. The pH was varied in the range of 2–12. Fig. 2 shows the results of S/Hg molar ratios of 0 and 1. When the S/Hg molar ratio is 0, 100% of the mercury exists as Hg(II) at pH = 2. The mercury concentration gradually goes down due to the precipitation of HgO. When the S/Hg molar ratio is 1, essentially all of the Hg is in the form of insoluble mercury-sulfide complexes, and the total dissolved mercury concentration is extremely low. The solubility of HgS is somewhat sensitive to pH. The lowest solubility happens over the pH range of 4–6. The solubility increases at both low and high pH values.

3.1.1. Batch kinetic studies

The kinetics of the adsorption of mercury on PAC and PAC-s are shown in Fig. 3. The Y-axis of the figure is the percentage adsorption of Hg²⁺,

$$\% \text{ Adsorption} = \frac{C_0 - C_e}{C_0} \times 100$$

where C_0 = initial concentration of mercury(II) and C_e = equilibrium concentration of mercury(II). Within 10 min, 87% of the mercury was adsorbed onto the PAC-s from the Hg solution, while 73% of the mercury was adsorbed onto the PAC-s from the surrogate within 30 min. The slower rate of adsorption for the surrogate is possibly due to the required release of mercury from the surface of the sand. At 24 h, nearly 99.9% of the mercury was adsorbed for both the Hg solution and the surrogate. No significant adsorption was observed after 24 h. Sen and De [5] obtained an equilibrium time of 30 min for activated carbon when they tried to remove mercury from wastewater. The difference between the equilibrium time

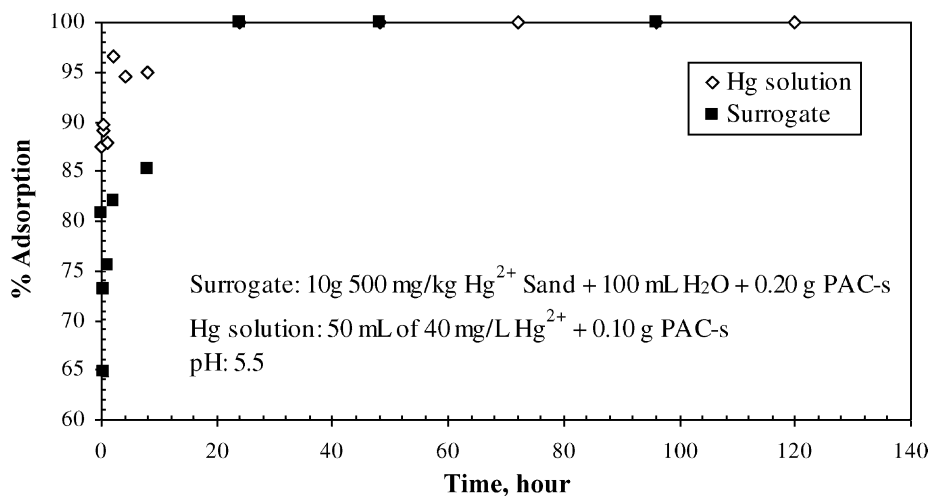


Fig. 3. Batch kinetic studies.

obtained by Sen and De [5] and this study could be due to the difference in composition of the activated carbons used. The activated carbon used by Sen and De [5] was a microcrystalline form of graphite, where more uniform diffusion may be possible. The activated carbon used for the batch kinetic studies in this research was thermally reactivated, and then treated by soaking in CS_2 . As a result, uniform diffusion of adsorbate in the adsorbent may not be possible. Moreover, the reaction between sulfur on the carbon surface and mercury species may also affect the rate of adsorption.

Figs. 4 and 5 show the effect of pH on carbon adsorption of mercury from mercuric nitrate solutions and from the surrogate, respectively. Apparently, sulfur-treating the PAC, greatly improved the mercury adsorption capacity, possibly due to the formation of mercuric sulfide on the surfaces of the carbon [8]. The figures show that adsorption on PAC-s is more sensitive to pH than on PAC. This may be due to the reaction between mercury and sulfur. A similar trend can be observed in the HgS solubility simulation (Fig. 2). Generally, the equilibrium mercury(II) concentration decreased with increasing pH up to a certain range, and then increased with further increase in pH. The optimum pH for Hg sorption was found to be in the pH range of 5.0–5.5 for both the Hg solution and the surrogate, when PAC-s was used.

3.1.2. Effect of initial concentration of mercury on the adsorption

Fig. 6 shows the effect of initial concentration of mercury on the adsorption. The percentage adsorption of mercury(II) at higher initial mercury concentrations was lower than that at lower initial concentrations of mercury(II). At low concentrations (below 500 mg/kg for 0.50 g PAC-s per 10.0 g surrogate or 100 mg/kg for 0.20 g PAC-s per 10.0 g surrogate), 100% adsorption occurred. This suggests that PAC-s can stabilize almost all mercury(II) in the surrogate if its mercury(II) concentration is below 500 mg/kg (0.50 g PAC-s per 10.0 g surrogate) or 100 mg/kg (0.20 g PAC-s per 10.0 g surrogate). Even for the surrogate with

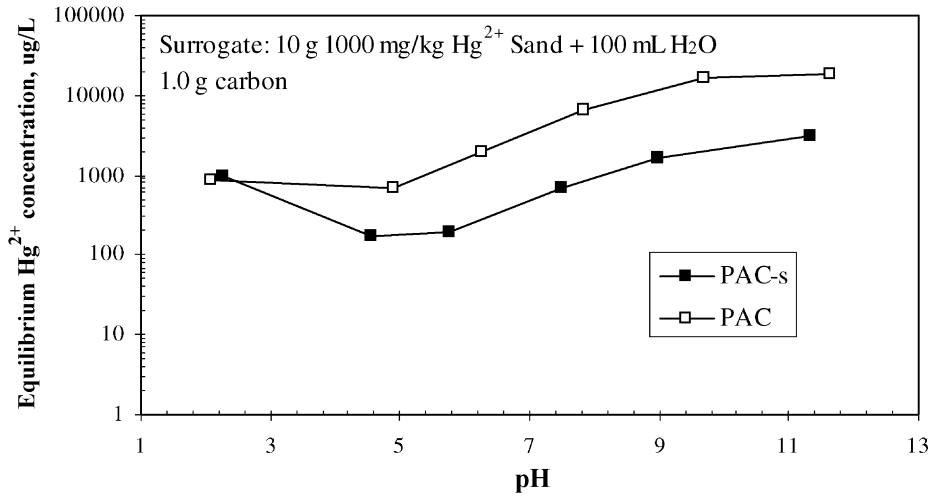


Fig. 4. Effect of pH on adsorption of mercury in the surrogate.

1000 mg/kg mercury(II), mercury(II) was very well stabilized: 96% and over 99% were adsorbed for the 0.20 g and 0.50 g PAC-s per 10.0 g surrogates, respectively.

Fig. 7 shows the TCLP results for the PAC-s stabilized surrogates. The control samples used in this figure were 5-day OPC-solidified surrogates with various initial mercury(II) concentrations (without activated carbon stabilization). It can be seen that the amounts of mercury leached out from all stabilized surrogate samples were well below the TCLP limit, which is 0.20 mg/l. This indicates that mercury(II) is strongly held onto PAC-s.

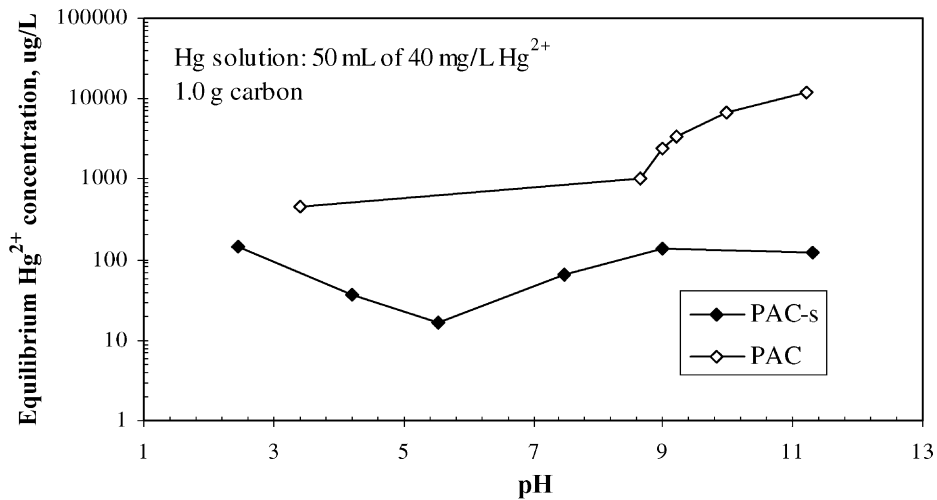


Fig. 5. Effect of pH on adsorption of mercury in the mercury solution.

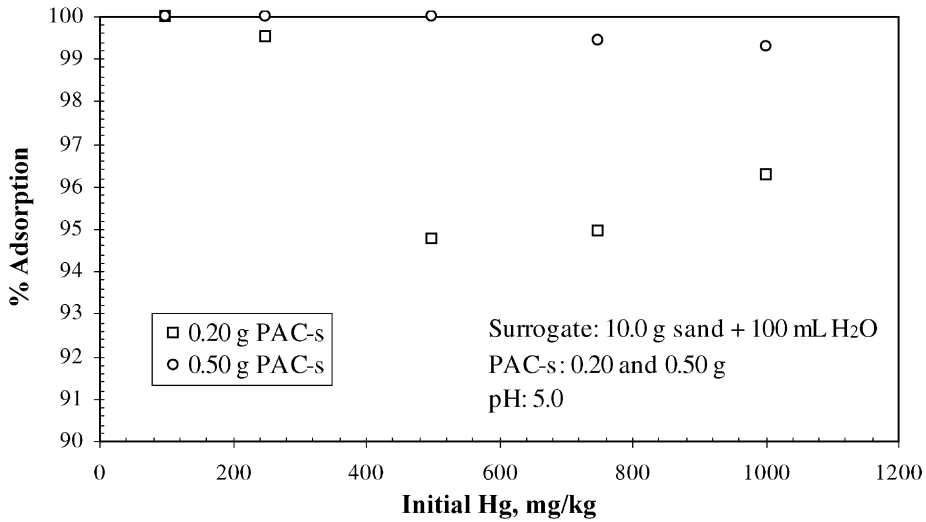


Fig. 6. Effect of initial mercury concentration on adsorption.

3.1.3. Effect of PAC-s dosage on the adsorption process

The effect of PAC or PAC-s dosage on the stabilization of mercury(II) in the surrogate is presented in Fig. 8. With increasing PAC dosage from 0.5 to 20% w/w, the adsorption percentage of mercury gradually increased from 31.0 to 98.5%. However, PAC-s showed a very different trend. Even for a very low dosage of PAC-s (0.5%), the adsorption percentage of mercury was very high (95.6%). With increasing PAC-s dosage, the mercury adsorption percentage quickly increased to over 99%.

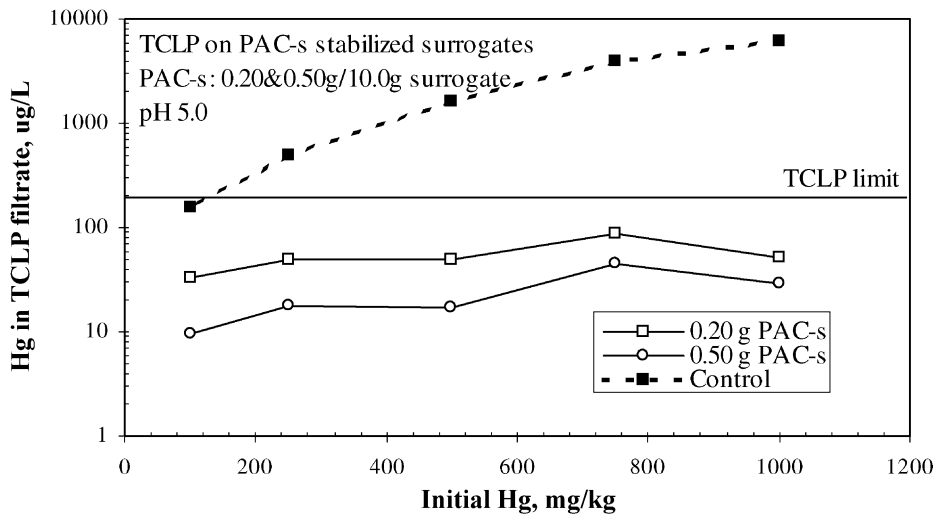


Fig. 7. TCLP results of PAC-s stabilized surrogates with various initial mercury concentrations.

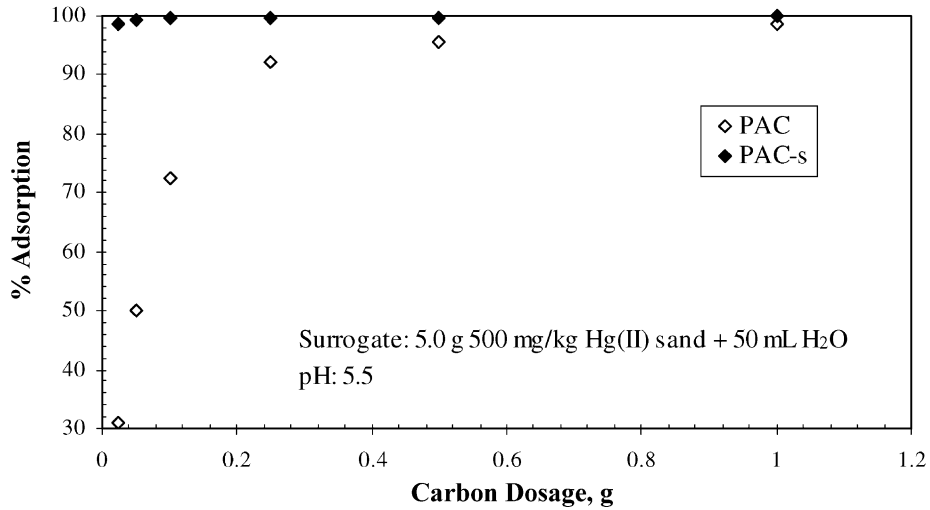


Fig. 8. Effect of carbon dosage on adsorption of mercury in the surrogate.

Adsorption of mercury(II) on PAC and PAC-s was found to conform to the Freundlich adsorption isotherm,

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log C_e$$

where x is the amount of mercury adsorbed (mg), m the amount of carbon used (g), k and $1/n$ empirical constants, and C_e is the equilibrium concentration of mercury (mg/l). The Freundlich isotherms for PAC and PAC-s are presented in Fig. 9. The Freundlich adsorption

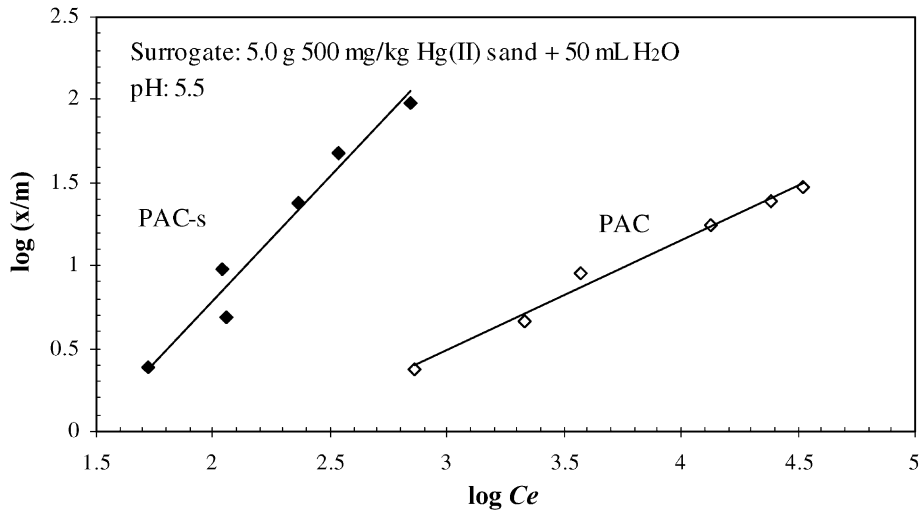


Fig. 9. Freundlich isotherms of mercury adsorption.

Table 2
Freundlich adsorption parameters of PAC and PAC-s

Adsorbent	k	$1/n$	Correlation coefficient
PAC	1.489	0.6596	0.989
PAC-s	2.173	1.4843	0.962

parameters k and $1/n$ are given in Table 2. The values of k and $1/n$ for PAC-s are higher than those for PAC, which indicates that mercury adsorption capacity of PAC-s is higher than that of PAC.

3.1.4. Interferences

The effect of interferents on mercury S/S by PAC-s and OPC was investigated by introducing the following anions and cation into the experiments: Cl^- , PO_4^{3-} , Ac^- , SO_4^{2-} , CO_3^{2-} , and Pb^{2+} . Fig. 10 shows the effect of Cl^- and PO_4^{3-} on the adsorption process. PO_4^{3-} and other ions (not shown in the figure) had little or no effect on mercury stabilization by PAC-s, but Cl^- had a strong influence on this process. With increasing initial mercury concentration or Cl^- concentration, the mercury concentration in the stabilization solution increased. This phenomenon has been studied by several researchers [18–20]. The increase of mercury release with increasing Cl^- concentrations is attributed to the dissolution of the adsorbed mercury through its complexation with Cl^- . At a Cl^- concentration of 0.1 mM (naturally occurring), the solubility of HgS increases by a factor of 408 [18]. However, the mercury stabilization process using PAC-s was still effective. Even at the Cl^- concentration of 1 mM, which is 10 times higher than the natural Cl^- concentration, the mercury released from a surrogate containing 1000 mg/kg mercury and stabilized with PAC-s was relatively low, only 1.63 mg/l.

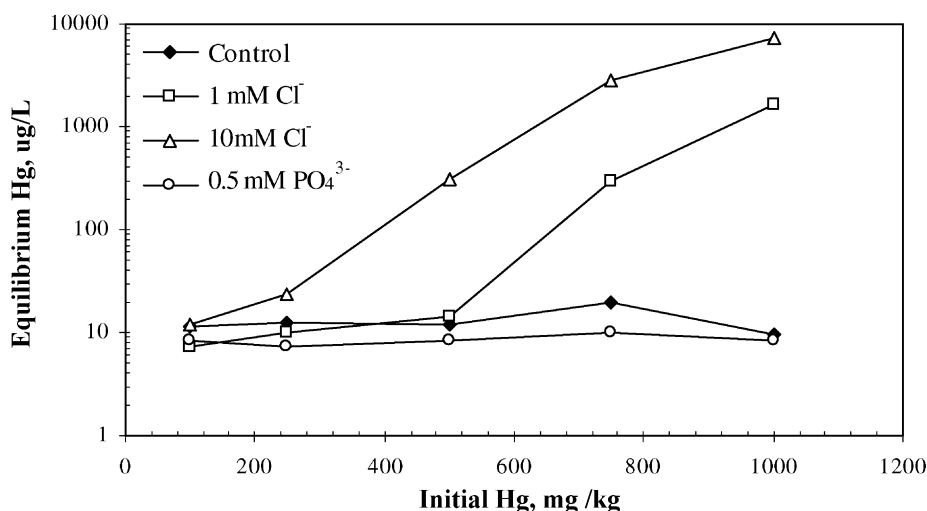


Fig. 10. Effect of interferents on adsorption of mercury in the surrogates.

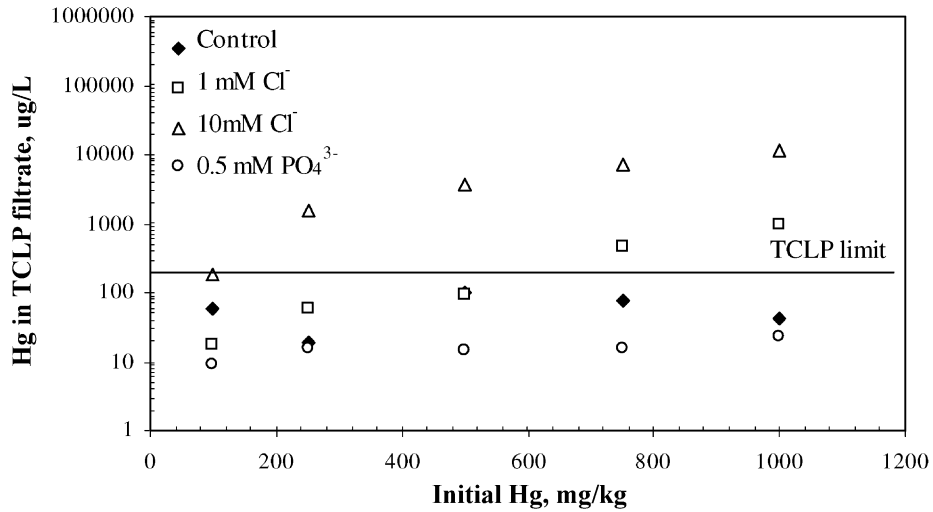


Fig. 11. Effect of interferents on TCLP results of PAC-s stabilized surrogates.

The stabilized surrogates were subjected to the TCLP leaching test (Fig. 11). PO_4^{3-} and other ions (not shown in the figure) had little effect on TCLP leaching results. However, all samples failed the TCLP test at a very high Cl^- concentration of 10 mM. At a Cl^- concentration of 1 mM, the stabilized surrogate started to fail the TCLP test when the initial mercury concentration was higher than 750 mg/kg. Considering that the acetate used in the TCLP test was found to have little interference for mercury adsorption, it is possible

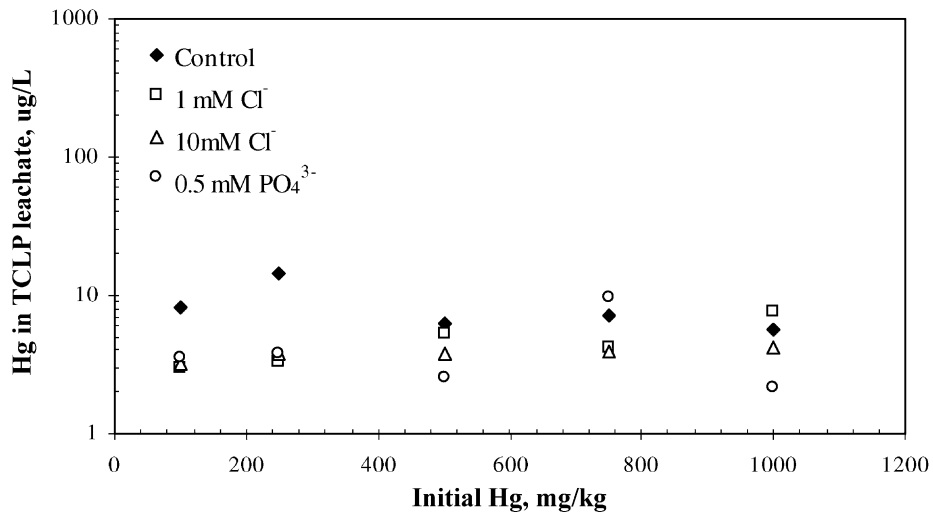


Fig. 12. Effect of interferents on TCLP results of PAC-s and S/S treated surrogates.

that chloride ions and mercury-chloride complexes remaining on the surrogates caused the failure of the TCLP test.

The stabilized surrogates were also solidified using OPC. After setting for 5 days, the cement paste mixture was crushed and subjected to the TCLP test. Fig. 12 shows the TCLP test results for the crushed samples. All samples leached very little mercury inspite of the presence of chloride. Arafat [21] found that encapsulation of carbon particles in cement, greatly reduced the desorption of phenol from reactivated carbon. In fact, as the hydration time exceeded 14 h, the amount desorbed was almost zero. It was postulated that, as the hydration of cement proceeds, there is a build-up of a gel-membrane outside the carbon pores. This indicates that, once in the solidified waste form, activated carbon particles will retain most of the adsorbed mercury, even in the presence of high concentrations of chloride ion.

4. Conclusions

From the results presented above, it is evident that stabilization of mercury in solid wastes by reactivated carbon is successful. Pretreatment of the PAC by soaking it in CS_2 effectively improves the mercury adsorption capacity of the PAC, possibly due to formation of mercuric sulfide on the activated carbon surface. The optimum pH for the mercury stabilization using PAC is in the range of 5.0–5.5. The adsorption equilibrium can be reached within 24 h. The adsorption of mercury by PAC and PAC-s are in accordance with Freundlich theory. In the study of stabilization of the surrogates without interferences, mercury-loaded PAC-s successfully passed the TCLP leaching test.

Only chloride ion had a significant effect on mercury adsorption by PAC or PAC-s among all the anions and cations investigated. OPC solidification of PAC-s stabilized surrogates significantly reduced interference by the chloride ion, by forming a barrier outside of the activated carbon particles. It can be concluded that the stabilization/solidification process using PAC-s and OPC is an effective technology to treat and dispose mercury-containing solid wastes. Compared with other processes, such as widely used sulfide precipitation, this technology is cleaner and more effective.

Acknowledgements

The authors are thankful to Calgon Carbon Company for providing the reactivated carbons for this study.

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