Degradation vs. Entrapment of PAHs and PCBs During Slurry Treatment with Hydrated Lime and Lime-Activated Persulfate

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INTRODUCTION

Results from recent pilot-scale studies indicate that adding quicklime (CaO) (also known as calcium oxide) reduces concentrations of PAHs and PCBs. However, the literature provides no clear mechanism for the alkaline destruction of PAHs and PCBs. In fact, the literature has shown that PCBs and PAHs are merely encapsulated by entrapping the contaminants in a coating of precipitated $Ca(OH)_2$, which is produced from the reaction of quicklime with water (Croft, 1967; Conner, 1993; Yilmaz et al., 2003). If this is the case, reducing the pH after apparent contaminant reductions with lime treatment would dissolve $Ca(OH)_2$ and release the PAHs and PCBs. However, the method for extracting PCBs and PAHs in soil samples (US EPA SW846 Method 3545) post-lime treatment (i.e. pH>11) does not require that the pH be lowered to circum-neutral values first.

The reaction of quicklime with water is shown below. This reaction is exothermic, and produces hydrated lime, or calcium hydroxide ($Ca(OH)_2$). The heat generated from this reaction, also known as lime slaking, generates significant amounts of steam and off-gassing. In studies of quicklime treatment of soils contaminated with PCBs (US EPA, 1991, Dàvila et al., 1993) and petroleum hydrocarbons (Shifano et al., 2007), the reduction in contaminant concentrations observed has been shown to be almost completely due to volatilization caused by the heat generated.

$$CaO_{(s)} + H_2O \rightleftharpoons Ca(OH)_{2\,(s)} \qquad (\Delta H_r = -63.7 \text{ kJ/mol of CaO})$$

Because of the inability of quicklime to actually degrade organic contaminants, a limited number of studies can be found in the literature about quicklime remediation of soils contaminated with organics. A laboratory study on the treatment of PCB contaminated soils with quicklime has been carried out by US EPA (1992). Temperatures of 180–200 °C were measured in the soils after mixing them with quicklime. PCB removal was mostly attributed to dusting, vaporization and steam stripping, and about 7% of the removal was attributed to de-chlorination. Marion and Payne (1997) and Marion et al. (1997) evaluated the mixing of quicklime for remediating soils contaminated with pesticides and petroleum hydrocarbons. The soils were mixed with quicklime in large on-site treatment units. The authors observed a major decrease in the concentration of many hydrocarbons. They explained the removal of volatile BTEX constituents as due to volatilization and that of higher molecular weight constituents as the result of encapsulation in a Ca(OH)2 matrix. Dean (2003) mixed quicklime to treat about 10,000 cubic yards of deep clayey soils. Trichloroethene and 1,1,1-trichloroethane soil concentrations were reduced to below the leachability soil cleanup target levels.

Even though there is no mechanism for the degradation of PCBs and PAHs with quicklime, it is still used to treat some soils, especially those with mixed inorganic (e.g., metal) and organic contamination (Conner, 1993; Yilmaz et al., 2003). Recently Waisner et al. (2008) reported PCB and PAH "removal" in soils treated with quicklime. However, did not adjust the pH before extraction. One possible mechanism for the entrapment of organic contaminants with lime is the coating of solid surfaces with hydrated lime $(Ca(OH)_2)$. Hydrated lime is relatively insoluble, with a solubility product of only 5.02×10^{-6}. Bonan (2005), using back-scatter electron microscopy, showed that $Ca(OH)_2$ precipitates onto solid surfaces (irrespective of mineral type) in layers 2-3 μm thick. Because solid surfaces tend to adsorb hydrophobic organics like PCBs and PAHs, these surfaces can get coated with $Ca(OH)_2$, which could interfere with organics from being extracted using EPA Method 3545.

The purpose of these studies was to determine whether PCBs and PAHs in a soil and a sediment are actually degraded by the addition of lime, or whether these compounds are merely entrapped by $Ca(OH)_2$ precipitation. In order to avoid the effect of heat generated by quicklime, $Ca(OH)_2$ was added directly as the soil amendment.

MATERIALS & METHODS

Test Soil and Sediment

Both a sediment and a soil contaminated with PAHs and PCBs were used in these studies. See Table 1 for the characteristics of the soil and sediment. The sediment was obtained from the Kalamazoo River during dredging operations. The soil was from a treatment facility for excavated soils in Montreal, Canada. Naphthalene (the most volatile PAH) represents less than 2% of the total PAH concentration in both the soil and sediment. The main difference between the sediment and soil is that the sediment has a high native organic matter (NOM) content (11%) whereas the soil has a low NOM content (<0.2%). The soil and sediment were first mixed for 24 hours in a 80-L cement mixer to homogenize them for the experiments.

Table 1. Properties of the homogenized sediment and soil used in the studies.

Characteristic	Sediment	Soil
pH	6.9	7.3
Native organic matter (NOM) content (%)	11%	<0.2
Sand (%)	23	91
Silt (%)	19	7
Clay (%)	58	2
PCB concentration (mg/kg)	6445 ± 761[a]	3847 ± 469
PAH concentration (mg/kg)	8301 ± 829	6602 ± 620

[a] mean ± standard deviation.

Lime and NaOH Dosing Experiments

The lime dosing experiments began on December 12th, 2009. They were done in 500 mL Erlenmeyer flasks shaken continuously on a shaker table. Each flask received 100 g of the homogenized soil, and was then filled to the 200 mL mark with DI water, or with a 20% sodium persulfate solution made in DI water. This resulted in a slurry with a solids concentration of 50% (w/v). A pH value above 11 is recommended for lime stabilization of soils and sediments (Conner, 1993), and a minimum pH of 10.5 is required to achieve alkaline activation of persulfate. The dosing-pH tests with and without persulfate were kept for a period of 3 weeks. This was done in order to give the persulfate a chance to activate, which lowers the pH by producing sulfuric acid. Each day the water level was adjusted with DI water to compensate for evaporation. The pH measurements were made with a pH probe inserted into the slurry.

A separate set of dosing experiments using NaOH were also done to compare with the hydrated lime dosing experiments for the soil and the sediment. The reactors, slurry concentration, and mixing were identical to those used in the lime dosing experiments, with the sole exception that 10 M NaOH rather than hydrated lime was added to increase the pH. A test reactor was set up with both the soil and the sediment. The pH was adjusted to above 11 by adding 10 M NaOH. The pH of the slurry was checked every 6 hours and every day thereafter. Additional NaOH was provided as necessary to keep the pH above 11, since the buffering capacity of the soil acted to decrease the pH.

After the 3 weeks of maintaining the pH above 11, the NaOH slurry and selected vessels from lime dosing studies were sacrificed to measure PCB and PAH concentrations. The slurries of soil and sediment with no lime added were used as a control. The slurries with lime amendments having final values of pH > 11 for each category tested (i.e., soil and sediment, with and without persulfate) were first combined into a 1 L Erlenmeyer and thoroughly mixed. There were three Erlenmeyer flasks having a final pH greater than 11 for the following four categories; soil without persulfate, sediment without persulfate, soil with persulfate, and soil with persulfate. The combined slurries were then split in

half. One half was used to extract and measure PCB and PAH concentrations without adjusting the pH, and the other half was first adjusted to a pH of 6 using 10 M HCl.

Slurry Reactors

The reactor vessels were 5 L closed glass reaction vessels with a mechanical mixer and multiple sampling ports in the lid (Figure 1). These sampling ports were used to measure pH, temperature, and to take slurry samples for monitoring contaminant concentrations. One of the ports housed two Tenex[®] activated carbon tubes in series to quantify off-gassing of PAHs and PCBs. The mechanical mixers were run continuously at approximately 350 rpm. For both the sediment and the soil, three reaction scenarios were tested; control (nothing added), hydrated lime addition, and persulfate with hydrated lime addition. In total, 6 slurry reactors were maintained, as listed and described in Table 2.

Table 2. The six slurry reactors tested in these studies.

Reactor Name	Amendments
Soil-Control	Nothing added
Sediment-Control	Nothing added
Soil-Lime	2% Ca(OH)$_2$ by weight
Sediment-Lime	2% Ca(OH)$_2$ by weight
Soil-Lime/Persulfate	3% Ca(OH)$_2$ by weight + 20% persulfate conc.
Sediment-Lime/Persulfate	3% Ca(OH)$_2$ by weight + 20% persulfate conc.

The reactors without persulfate were prepared by adding 2 kg of soil or sediment (dry weight) and then enough DI water to reach the 4 L mark. Finally, the dose of lime was added and the rectors were closed. Reactors with persulfate were prepared the same way except that 646 g of sodium persulfate were added before the 3% lime dose. This resulted in a sodium persulfate concentration of 20%.The pH was measured with a probe.

The slurry reactors were started on January 9th and were maintained for 22 days. Based on the dosing studies, a Ca(OH)$_2$ dose of 2% was used in the reactors without persulfate and 3% was used in the reactors with persulfate. All PAH and PCB samples were taken in triplicate allowing a standard deviation to be obtained for each data point.

Figure 1. Picture of the closed slurry reactor set-up, showing the mechanical mixer, the lid with ports, and the two activated carbon traps in series.

Sampling and Analytical Methods

Triplicate 20 mL slurry samples were taken to quantify PAHs and PCBs. The soil pellet, containing 10 g (dry weight) of soil, was first spiked with 0.1 mL of a 2% solution of 2-fluorobiphenyl in hexane and thoroughly mixed to allow recovery to be quantified. The soil pellet was then extracted for PCBs and PAHs using EPA Method 3545 without pH adjustment (pH>11). A second set of triplicate slurry samples was first adjusted to a pH of 6 using HCl before being centrifuged and extracted using EPA Method 3545. A volumetric ratio of hexane/acetone of 1/1 was used for extraction. The extract was then concentrated to volume of 5 mL, and was then divided in two: one half for PCB analysis and the other for PAH. The extract for PCB analysis was first cleaned according to EPA Method 3665A using sulfuric acid, and was then analyzed using a Hewlett-Packard 5890 GC equipped with electron capture detection (ECD) according to EPA Method 8082. The extract for PAH analysis first cleaned using silica gel using EPA Method 3630C. The cleaned extract was then analyzed for PAHs using a Hewlett-Packard 5890 GC/MS according to EPA Method 8270C for SVOCs. Recovery of 2-fluorobiphenyl was never below 86%, which indicates that the extraction of PCBs and PAHs was effective. The persulfate anion was also quantified in filtrate (0.25 μm) samples using the iodometric titration method (Kolthoff and Stenger, 1947). Temperature and pH were

RESULTS

Lime Dosing Results

The results of the lime dosing studies without and with persulfate are shown in Figures 2 and 3, respectively. The presence of 20% persulfate increased the amount of hydrated lime necessary to increase the pH to above 11 (Figure 3) relative to slurry with no persulfate (Figure 2). Based on these studies, the dose of hydrated lime chosen for the slurry reactor studies was 2% without persulfate, and 3% with the 20% persulfate.
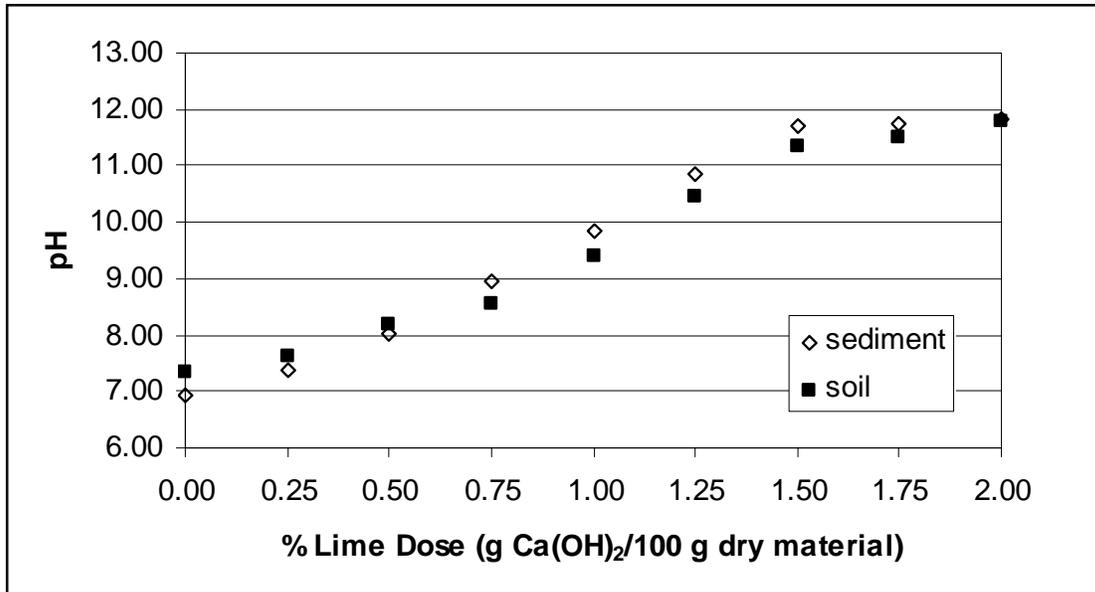
Figure 2. The effect of hydrated lime dose on pH in slurries of the soil and sediment without persulfate.
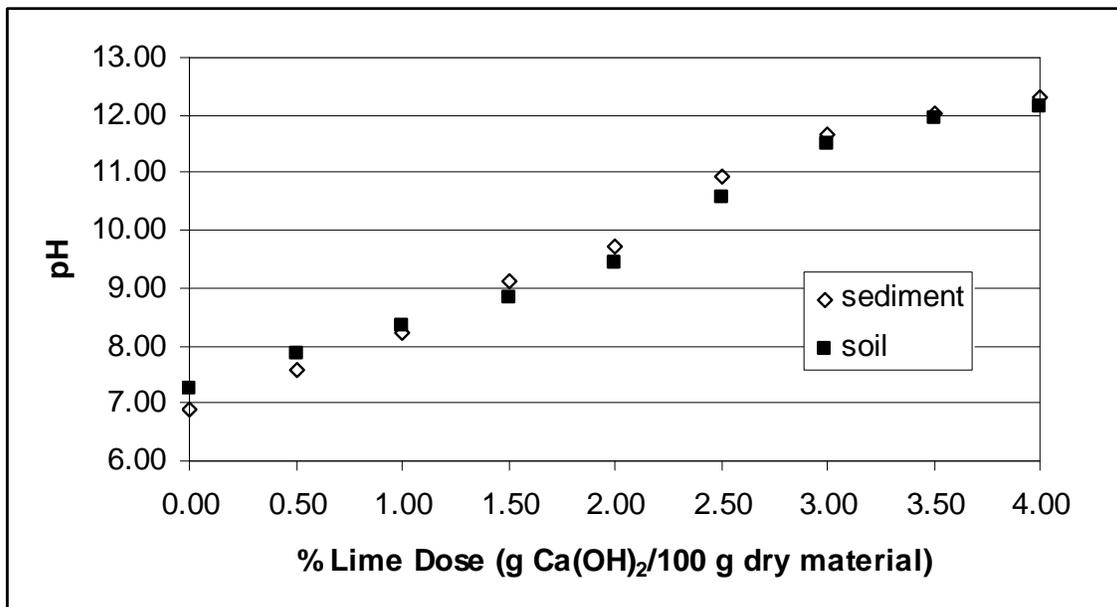

Figure 3. The effect of hydrated lime dose on pH in slurries of the soil and sediment having a 20% persulfate concentration.

Contaminant Measurements with Lime and NaOH Dosing

Contaminant measurements were made after 3 weeks of maintaining a pH above 11 with either lime or NaOH. Although slurries contain more water than soil piles or in situ soil, the extra water in a slurry allows for excellent mixing, and makes pH measurements easy and accurate. Measuring pH in solids requires mixing water with the solids (2:1, vol/vol) and measuring the pH of the resulting slurry. Moreover, it was necessary to have a slurry in order to measure persulfate concentrations in the water.

After 3 weeks of the lime dosing studies, the flasks containing persulfate with a pH above 11 were tested. There was no persulfate remaining in any of them, demonstrating that the high pH promoted persulfate activation. The results from the contaminant measurements in the dosing studies are listed in Table 3. The reaction scenarios labeled "control" consisted of the flasks with no lime or NaOH added, and the other reaction scenarios consisted of the flasks with a lime or NaOH dose high enough to maintain a pH above 11 throughout the 3 week period. The NaOH dosing studies served as a control to compare contaminant measurements with those obtained from lime dosing. Note that the reaction flasks were open and volatile losses of PCBs and PAHs were not quantified.

Table 3. Concentrations and percent removals of PCB and PAH measured in slurries treated with lime, lime with persulfate, and NaOH to achieve a pH>11.

Reaction Scenario	PCB Conc.	% PCB Removal	PAH Conc.	% PAH Removal
Soil-Control	3847 ± 446^a	n/a	6602 ± 527	n/a
Soil-Lime	1149 ± 292	70	2118 ± 265	68
Soil-Lime: pH adjusted	3470 ± 421	10	6225 ± 479	6
Soil-Lime/Persulfate	914 ± 177	76	675 ± 106	90
Soil-Lime/Persulfate: pH adjusted	1705 ± 303	56	1906 ± 229	71
Soil-NaOH	3974 ± 509	-3	6273 ± 520	5
Soil-NaOH: pH adjusted	3861 ± 492	0	6419 ± 463	3
Sediment-Control	6445 ± 454	n/a	8301 ± 538	n/a
Sediment-Lime	2760 ± 189	57	3755 ± 363	55
Sediment-Lime: pH adjusted	6329 ± 393	2	7932 ± 562	4
Sediment-Lime/Persulfate	377 ± 89	94	1048 ± 119	87
Sediment-Lime/Persulfate: pH adjusted	1581 ± 190	75	2573 ± 203	69
Sediment-NaOH	6287 ± 561	2	8428 ± 633	-2
Sediment-NaOH: pH adjusted	6446 ± 489	0	8255 ± 569	1

[a] mean ± standard deviation for triplicate samples.

The results from Table 3 show that lime doses resulting in a pH greater than 11 significantly reduced the concentration of PCBs and PAHs measured in both the soil and the sediment. However, dropping the pH of the lime-treated soil and sediment before extracting for contaminants re-released the lime-entrapped contaminants. This effect was greatest in the slurries without persulfate, because the persulfate oxidized some of the contaminants. When NaOH was used instead of hydrated lime to raise the pH above 11, no significant reduction in PCB and PAH concentrations were observed in either the soil or the sediment. The results obtained with NaOH dosing show that the common method used for extracting PCBs and PAHs from soils and sediments (EPA method 3545) is appropriate for high pH soils that are not treated with lime. However, the difference in results obtained with added lime and NaOH suggest that entrapment caused by lime precipitation (or "lime-lock") was the predominant mechanism for the "apparent" reductions in concentrations of PCBs and PAHs in the lime-treated soils and sediments.

These results demonstrate that entrapment of contaminant can occur in lime-treated soils, and that pH adjustment of samples should take place before measuring organic contaminants such as PCBs and PAHs. Even though some lime-lock occurred with persulfate, as evidenced by the re-release of apparently degraded PCBs and PAHs after pH adjustment, persulfate oxidation did in fact achieve significant removal of PCBs and PAHs compared with lime treatment alone. In fact, the dose of lime in the persulfate flasks (3%) was 50% greater that in the flasks without persulfate (2%), which would increase the potential for lime-lock in the persulfate flasks.

The results from the lime dosing studies were important in determining the amount of hydrated lime required to maintain a pH above 11 in slurries of the test soils. In addition, the contaminant measurements provided preliminary evidence that lime-lock, or entrapment by lime precipitation of PCBs and PAHs, takes place in the test soil and sediment.

Slurry Reactors

The six slurry reactors contained 4 L of slurry having a 50% solids concentration (w/v). Based on the lime-dosing studies, a dose of hydrated lime of 2% was used for reactors without persulfate and a dose of 3% was used for reactors containing a 20% sodium persulfate concentration in the slurry water.

Figures 4 and 5 show the temperature and pH profiles of the slurry reactors serving as controls (i.e., no lime or persulfate added) for the soil and sediment, respectively. As is shown in the figures, the pH values remained near 6.9 for the sediment and 7.3 for the soil, which were the native pH values of these materials (Table 1). The temperature in both reactors remained at room temperature in both reactors (22-24 °C).
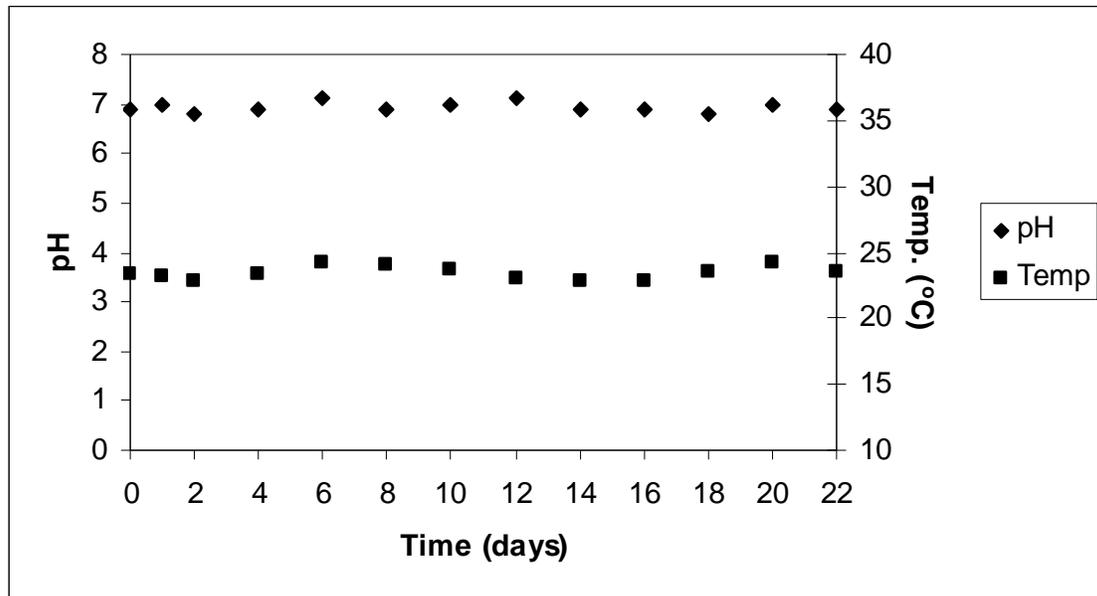
Figure 4. Temperature and pH profiles for the soil control (i.e., no lime added).
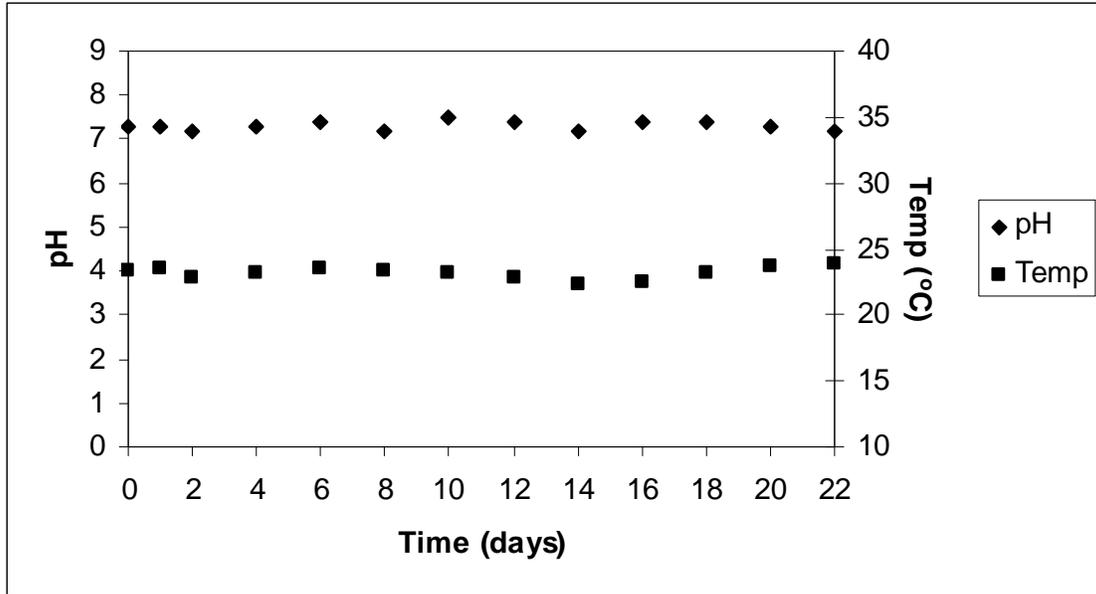
Figure 5. Temperature and pH profiles for the sediment control (i.e., no lime added).

Figures 6 and 7 show the temperature and pH profiles for the lime-treated soil and the lime-treated sediment, respectively. The pH of the both the soil and sediment remained between above 11 during the entire 22-day period, which indicates that the dose of hydrated lime determined in the lime dosing experiments (2% be weight) was appropriate. The temperature did not increase significantly above room temperature in either soil or sediment treated with lime, which is consistent with dosing with hydrated lime ($Ca(OH)_2$) vs. quicklime (CaO). This was done to avoid high temperatures associated with quicklime, which encourages off-gassing and volatilization.
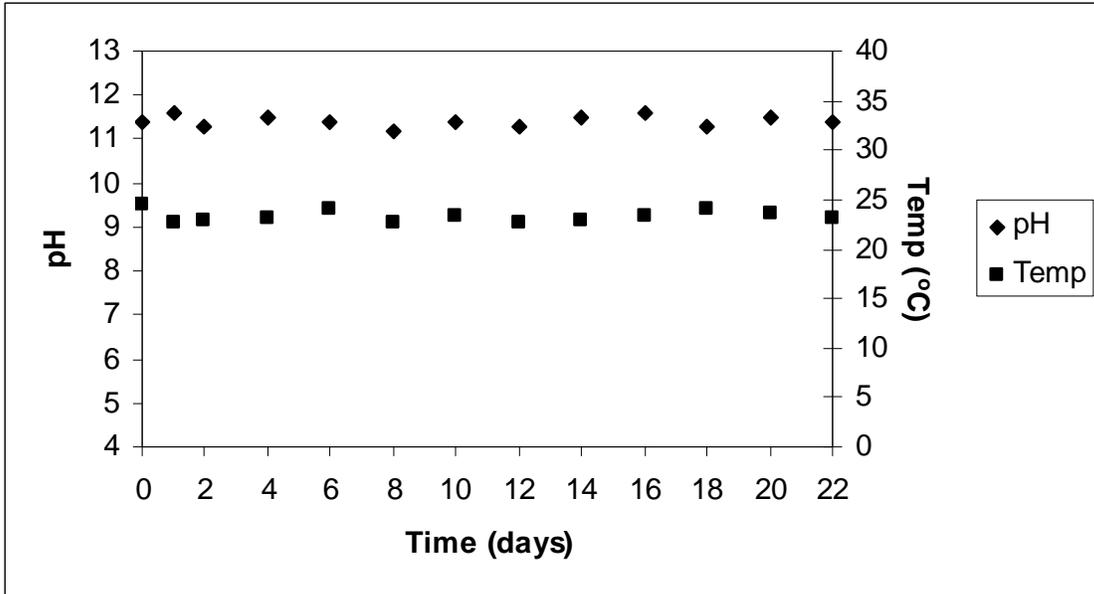
Figure 6. Temperature and pH profiles for the lime-treated soil.
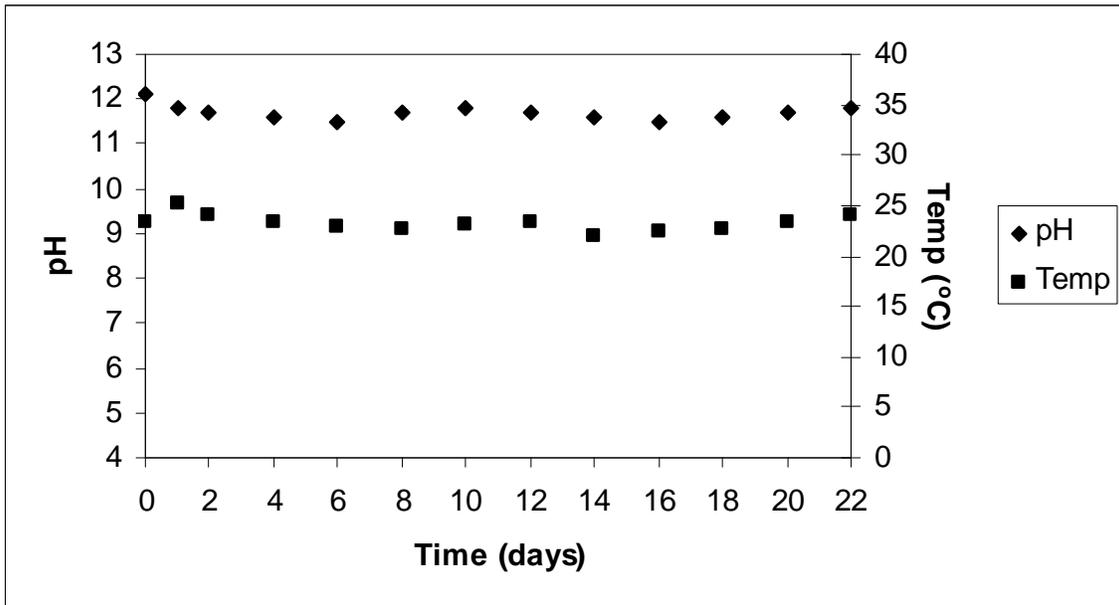

Figure 7. Temperature and pH profiles for the lime-treated sediment.

Figures 8 and 9 show the time profiles of temperature, pH, and persulfate concentrations in samples of slurry filtrate for the soil and sediment, respectively. The values of pH remained above 11 throughout study period in both the soil and the sediment. Figures 8 and 9 also show that the persulfate was completely activated within 16 days in the soil and 14 days in the sediment. This is consistent with the pH being above 11, as a minimum pH of 10.5 is required to activate persulfate. The temperature in both slurries undergoing lime/persulfate treatment increased significantly above room temperature during the period of persulfate activation (disappearance). Maximum temperatures observed were approximately 71 °C and 69 °C for the soil and sediment slurries, respectively. Elevated temperatures occur naturally during persulfate activation, which is an exothermic process.
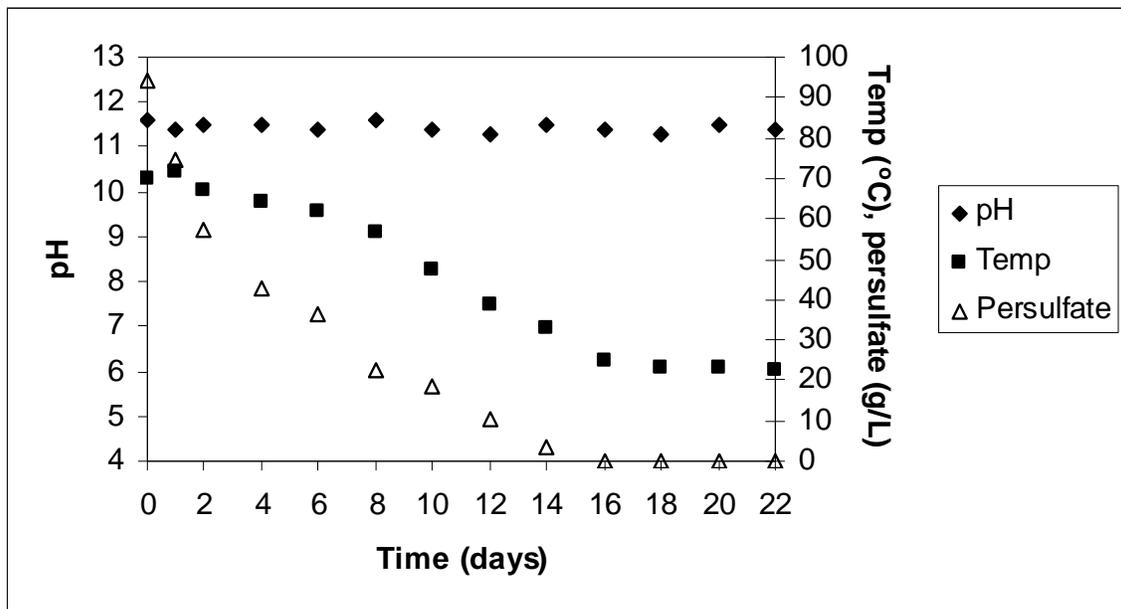

Figure 8. Profiles of temperature, pH, and persulfate concentrations for lime/persulfate treatment of the soil.
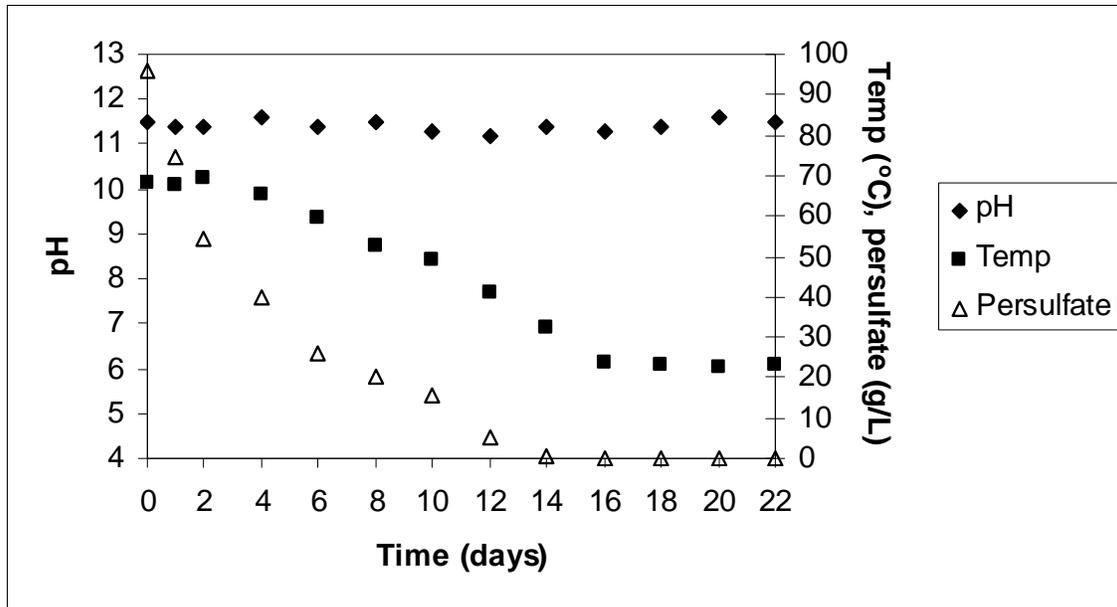
Figure 9. Profiles of temperature, pH, and persulfate concentrations for the lime/persulfate treatment of the sediment.

Figures 10 and 11 show the time profiles of the concentrations of PCBs and PAHs measured in the slurry reactors serving as controls (i.e., no lime or persulfate added) for the soil and the sediment, respectively. Both reactors maintained PCB and PAH concentrations that were statistically the same (i.e., within the standard deviation error bars) throughout the 22-day period, and were similar to the concentrations provided in Table 1. The results in Figures 10 and 11 indicate that these reactors proved to be appropriate controls to compare with lime and lime/persulfate treatment.
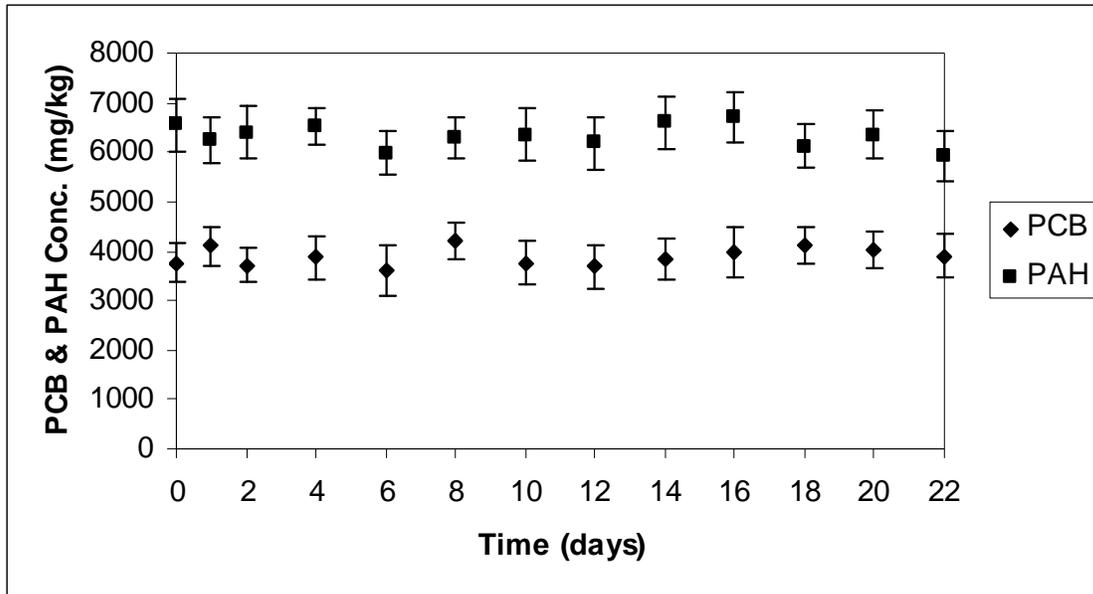
Figure 10. Time profiles of the concentrations of PCBs and PAHs measured in the soil control.
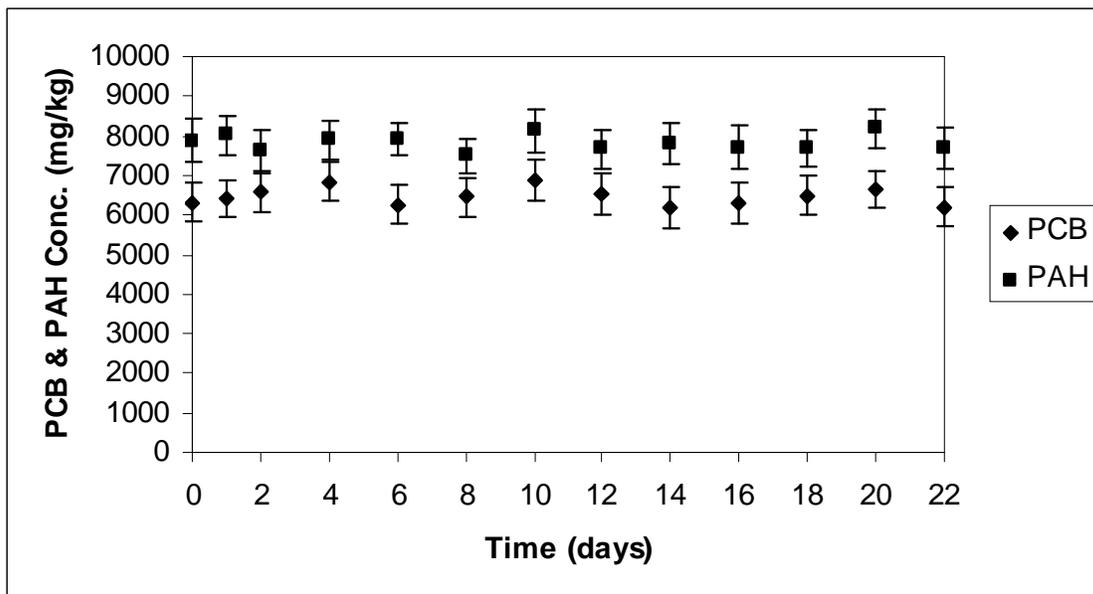

Figure 11. Time profiles of the concentrations of PCBs and PAHs measured in the sediment control.

Figures 12 and 13 show the time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for lime treatment of the soil and sediment, respectively. In both the soil and the sediment, pH-adjusted samples showed significantly higher concentrations of PCBs and PAHs than the samples extracted without pH adjustment. These results are consistent with the results obtained during the lime dosing studies, and support the hypothesis that lime-lock (i.e., entrapment of the contaminants within $Ca(OH)_2$ precipitate) occurred in the soil and sediment. It is clear that lime treatment does not degrade PCBs or PAHs, and that the extraction method commonly used for soils and sediments (EPA Method 3545) does not always extract all the PCBs and PAHs in such matrices subjected to lime treatment.

Figures 14 and 15 show the time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for lime/persulfate treatment of the soil and sediment, respectively. The pH-adjusted samples in soil and the sediment subjected to lime-activated persulfate treatment, showed only slightly greater concentrations of PCBs and PAHs than the samples at pH above 11. These results are consistent with those obtained during the lime dosing studies, and clearly show that significant chemical oxidation of PCBs and PAHs took place in the soil and the sediment. However, some entrapment or lime-lock of the contaminants did take place in the presence of persulfate. This observation also supports the use of pH adjustment prior to extraction of organic contaminant using EPA Method 3545 when the treatment method includes lime.
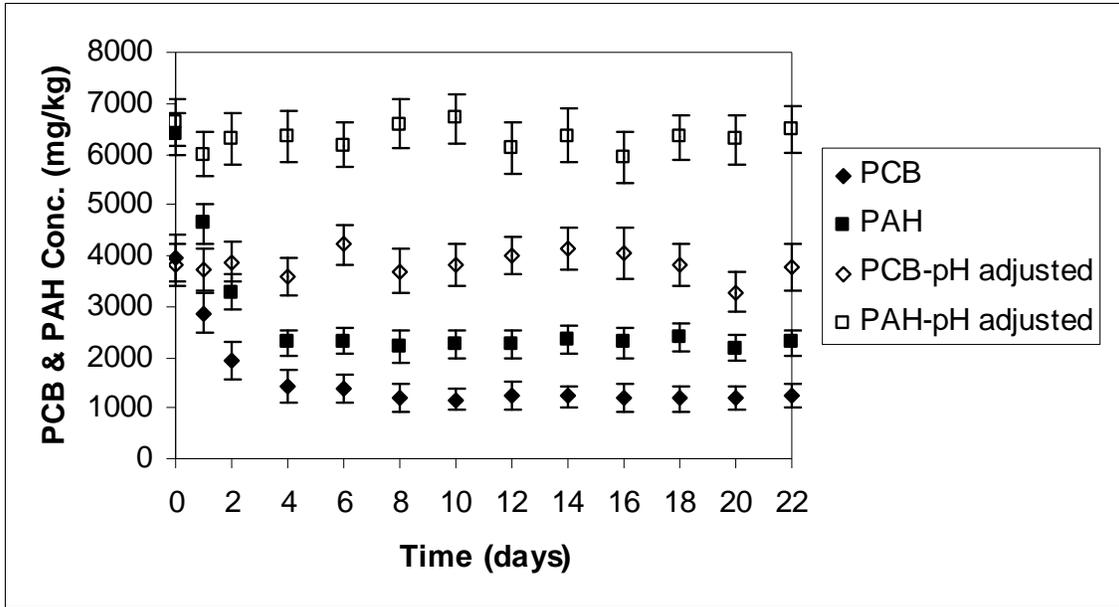
Figure 12. Time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for the soil treated with lime only.
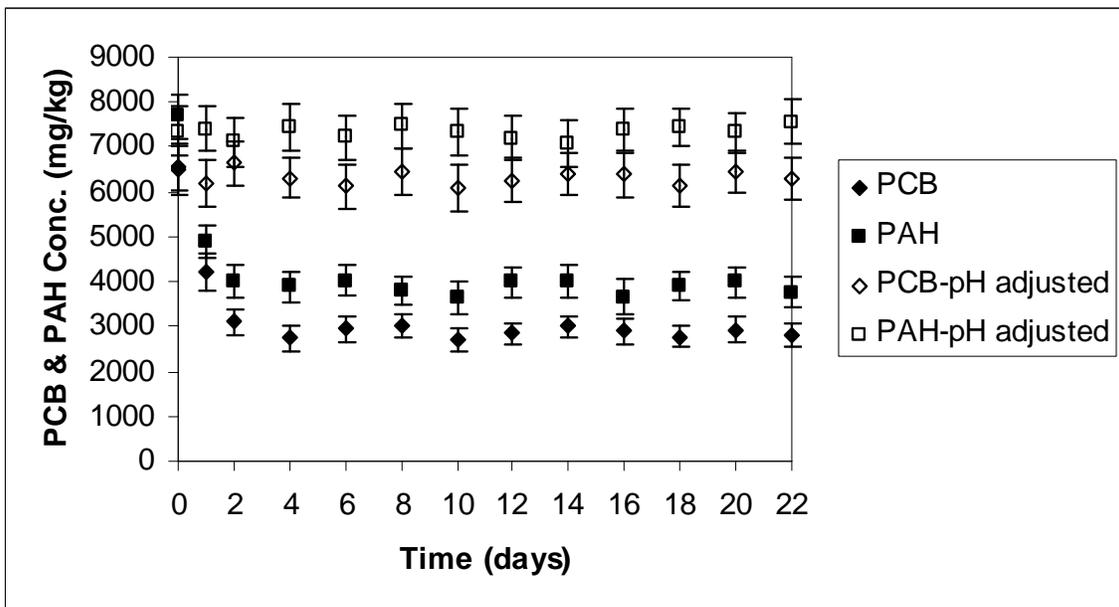

Figure 13. Time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for the sediment treated with lime only.
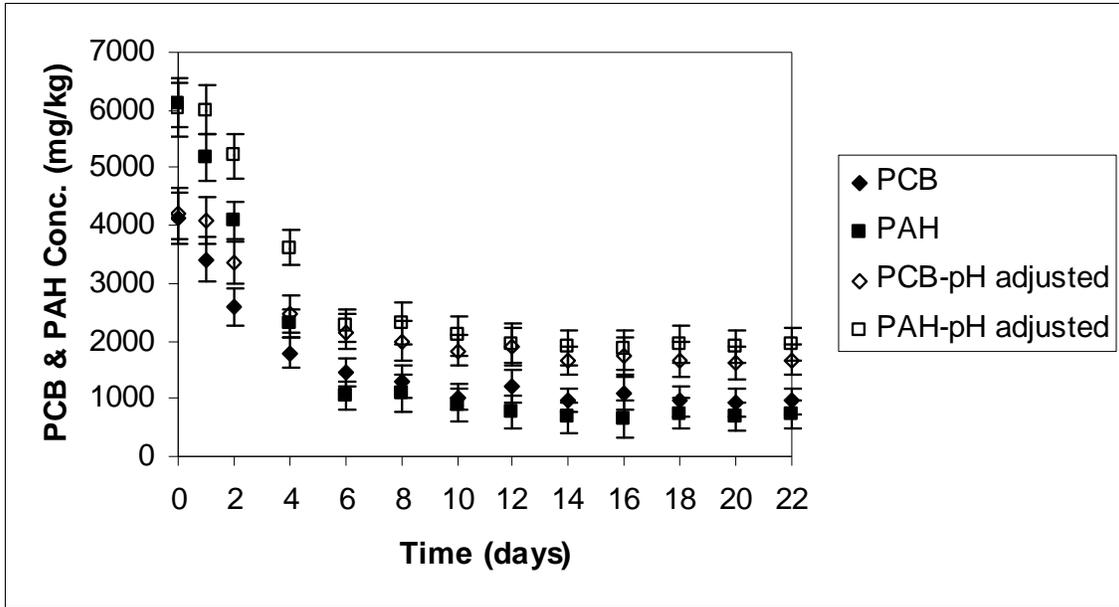
Figure 14. Time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for the soil treated with lime/persulfate.
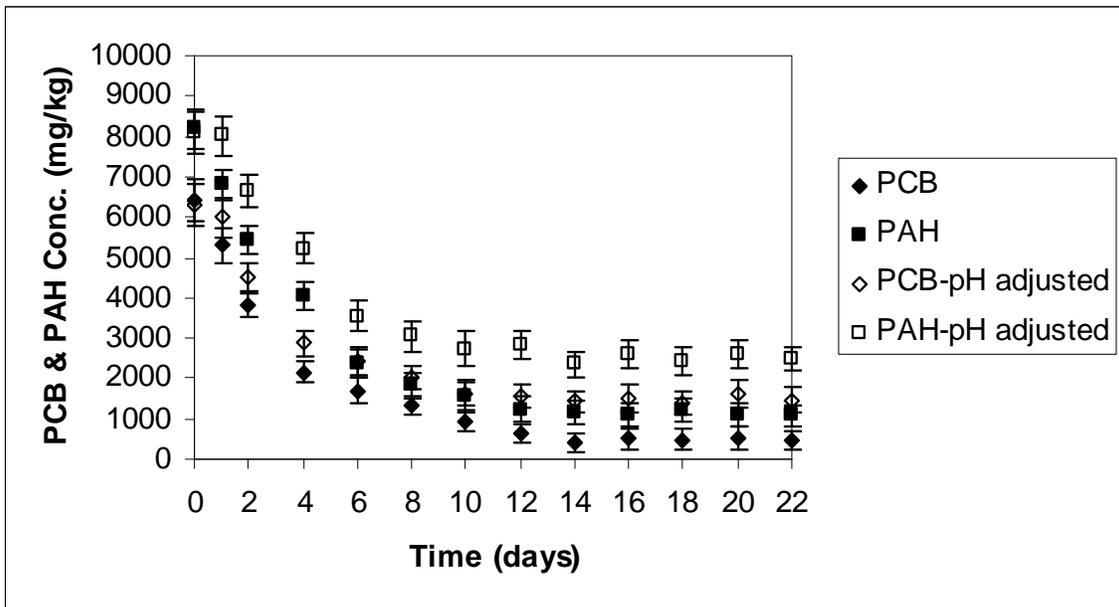

Figure 15. Time profiles of the concentrations of PCBs and PAHs measured before and after pH adjustment for the sediment treated with lime/persulfate.

Table 4 lists the initial and final concentrations of PCBs and PAHs in the slurry reactors for samples without and with pH adjustment, as well as the total percent removals and volatile percent removals measured for both contaminants. Note that there was more volatile removal in the persulfate reactors because the temperatures were higher than in the other reactors (see Figures 4 through 7). There was no volatilization of PCBs or PAHs detected in the lime-treated reactors, perhaps because of lime-lock caused by precipitated $Ca(OH)_2$, which would protect the organics from being volatilized.

Table 4. Results from the reactors showing the initial and final concentrations of PAHs and PCBs after 22 days, and the concentrations after adjusting the pH to 6.

Reaction Scenario	PCB	PAH	PCB with pH adjusted	PAH with pH adjusted
Soil-Control				
Initial concentration	3768 ± 401^a	6544 ± 514	n/a	n/a
Final concentration	3892 ± 433	5928 ± 522	n/a	n/a
Total Removal	-3%	9%	n/a	n/a
Volatile Removal	ND^a	0.6%		
Sediment-Control				
Initial concentration	6328 ± 502	7890 ± 532	n/a	n/a
Final concentration	6210 ± 482	7683 ± 504	n/a	n/a
Total Removal	2%	3%	n/a	n/a
Volatile Removal	ND	0.8%		
Soil-Lime				
Initial concentration	3940 ± 462	6392 ± 427	3812 ± 420	6631 ± 451
Final concentration	1241 ± 214	2282 ± 248	3786 ± 466	6492 ± 469
Total Removal	69%	64%	1%	2%
Volatile Removal	ND	ND		
Sediment-Lime				
Initial concentration	6539 ± 523	7680 ± 499	6483 ± 527	7340 ± 546
Final concentration	2814 ± 245	3768 ± 328	6287 ± 482	7560 ± 480
Total Removal	57%	51%	3%	-3%
Volatile Removal	ND	ND		
Soil-Lime/Persulfate				
Initial concentration	4121 ± 440	6117 ± 427	4208 ± 444	6019 ± 455
Final concentration	958 ± 213	739 ± 248	1661 ± 262	1941 ± 298
Total Removal	77%	88%	61%	68%
Volatile Removal	1.1%	2.4%		
Sediment-Lime/Persulfate	1.3%			
Initial concentration	6404 ± 523	8192 ± 502	6298 ± 531	8092 ± 532
Final concentration	467 ± 245	1091 ± 258	1470 ± 295	2493 ± 308
Total Removal	93%	87%	77%	69%
Volatile Removal	1.3%	2.1%		

[a] mean $\pm$ standard deviation for triplicate samples.
[b] ND=Non detect.

Lime treatment alone resulted in an "apparent" (i.e., non pH-adjusted) total removal of PCBs and PAHs in the soil of 69% and 64%, respectively. However, adjusting the pH to 6 caused those removals to drop to 1% for PCBs and 2% for PAHs. In the sediment, apparent total removals of PCBs and PAHs were 57% and 51%, respectively. However, adjusting the pH brought those removal numbers down to 3% for PCBs and -3% for PAHs. In contrast to lime addition alone, treatment with lime-activated persulfate showed significant values for total removal of PCBs and PAHs. In the soil, total PCB and PAH removals for pH-adjusted samples were 61% and 68%, respectively. The pH-adjusted samples in the sediment showed total removals of PCBs and PAHs of 77% and 69%, respectively. However, some lime-lock did occur with lime-activated persulfate treatment. The removals of PCBs and PAHs in samples above pH 11 were roughly 20% higher than in the pH-adjusted samples for slurries subjected to lime-activated persulfate.

CONCLUSIONS

Addition of hydrated lime to the test soil and sediment to achieve pH values above 11 resulted in "apparent" reductions in concentrations of PCBs and PAHs when the pH was not adjusted before extraction of samples with EPA Method 3545. However, adjusting the pH to a value of 6 before extracting the samples showed that the contaminants were merely prevented from being extracted. Soil and sediment treated to pH>11 with NaOH did not show significant reductions in PCBs or PAHs, demonstrating that a pH>11 does not destroy these contaminants, and that the high pH itself does not interfere with extraction using Method 3545. Rather, the reductions in PCB and PAH concentrations appear to be due to entrapment by precipitated lime. The proposed mechanism for this is entrapment of the contaminants under a coating of precipitated $Ca(OH)_2$, thereby protecting the contaminants from being extracted and measured. This phenomenon was also observed with 20% persulfate treatment, but the effect was quite muted compared with lime treatment alone. These results show that lime-activated persulfate treatment achieved significant destruction of PCBs and PAHs in the test soil and sediment, whereas lime treatment alone achieved little degradation of contaminants.

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