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NaOH Regeneration of Pb and Phenol-Laden Activated Carbon. I. Batch Study Results

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ABSTRACT

Granular activated carbon (GAC) has been used to remove organics and metals simultaneously from wastewaters (4). For a regeneration procedure to be effective on GAC containing both metals and organics, it must remove both the metals and organic compounds. In this study, GAC saturated with Pb and phenol was regenerated using an NaOH rinse in batch mode. Four variables were investigated: 1) regenerant concentration (0, 0.1, and 1.0 N), 2) regenerant to carbon mass ratio (5:1 and 10:1), 3) regenerant temperature (22 and 80°C), and 4) regeneration time (0.33, 1, 3, 7, and 21 days). Maximum regenerations of 47% for Pb and 48% for phenol were observed. The 1.0 N NaOH regenerated a higher percentage of both Pb and phenol when compared to the other regenerants. The 10:1 regenerant to carbon ratio resulted in increased regeneration over the 5:1 regenerations. Pb regeneration was slightly higher at 80°C than at 22°C whereas phenol regeneration was slightly lower at 80°C than at 22°C. Maximum regenerations for both Pb and phenol were observed within the first day of regeneration, in most cases within the first 8 hours.

INTRODUCTION

Activated carbon is an accepted method for the removal of organic contaminants. Recently, it has been demonstrated that activated carbon can also be used to remove Pb and phenol simultaneously from the aqueous phase (4). Regeneration of activated carbon laden with Pb has been accomplished using acid/base and base-only rinsing procedures (5). Batch kinetic studies were conducted to determine if a caustic rinse would be

effective in regenerating an activated carbon loaded with Pb and phenol. The following parameters were investigated: 1) NaOH concentration (0, 0.1, and 1 N), 2) temperature (22 and 80°C), and 3) regenerant:carbon mass ratio (5:1 and 10:1). Results from this study will be used in the design of a subsequent column study.

SELECTED BACKGROUND INFORMATION

Goto (2) and Rustamov (6) studied the regeneration of phenol-laden activated carbon using NaOH. Goto employed 0.5 N and 2 N NaOH and reported that the maximum phenol desorption was 70% and phenol adsorption gradually decreased with regeneration cycles (2). The initial phenol loading was 168 mg/g. Rustamov investigated different NaOH concentrations (1 to 15%) for phenol desorption (6). Phenol desorption was greatest (71%) at 5% NaOH. Both researchers postulated that the carbon-bound phenol reacted with Na to form the nonadsorbable C_6H_5ONa. Carbon-bound phenol may also deprotonate at higher pH values (pK_a for phenol is 9.9) with the ionic form of phenol being less adsorbable. Norit Americas, Inc. personnel reported that a client has successfully regenerated activated carbon that was loaded with phenolic compounds using a hot (80°C) caustic rinse (Personal Communication, 1995).

Reed et al. investigated the regeneration of Pb-laden GAC using 0.1 and 1 N NaOH (5). Regeneration efficiencies using 0.1 N NaOH averaged 47% while efficiencies at the higher NaOH concentration regeneration increased to 89%. The authors postulated that the carbon-bound Pb (adsorbed or surface precipitated) formed soluble species [e.g., $Pb(OH)_4^{2-}$] at high pH values. Pb removal in subsequent column runs decreased slightly between Runs 1 and 2 but stabilized between Runs 2 and 3. Pb regeneration efficiencies, using 1 N NaOH only, were similar to those observed when a HCl–NaOH serial rinsing procedure was used.

The simultaneous adsorption of Pb and phenol was investigated using Hydrodarco (HD) 4000 activated carbon (4). Seven treatment cycles were conducted. The regeneration procedure consisted of rinsing the GAC column with 10 bed volumes (BVs) of 0.1 N HCl followed by 10 BVs of either 0.1 or 1 N NaOH. The HCl rinse lowered the column pH, and the carbon-bound Pb was removed (cationic metal removal decreases with decreasing pH). Little phenol was removed during the HCl rinse, but large amounts of phenol were leached out of the column during the NaOH rinse. Phenol desorption was highest when 1 N NaOH was used. Optimization of the NaOH portion of the regeneration process (for phenol removal) was not extensively examined.

EXPERIMENTAL DESIGN

Batch kinetic studies were conducted to determine if a caustic rinse would be effective in regenerating an activated carbon that was loaded with Pb and phenol. The following parameters were investigated: 1) NaOH concentration (0, 0.1, and 1 N), 2) temperature (22 and 80°C), and 3) regenerant:carbon mass ratio (R:C, 5:1 and 10:1). Samples were removed from the shaking table and analyzed at 0.33, 1, 3, 7, and 21 days. In addition, phenol solutions with and without NaOH were heated to 80°C to determine the fate of phenol in a high pH and temperature environment. The activated carbon used in this investigation was Hydrodarco (HD) 4000, a lignite-based GAC manufactured by Norit Americas, Inc. (Atlanta, GA). Characteristics of HD4000, as supplied by the manufacturer, are presented in Table 1. Materials and methods are detailed in the following paragraphs.

TABLE 1
Characteristics of Hydrodarco 4000

Characteristic	Quantity
Base	Lignite
Type	L
Surface area (m^2/g)	625
Slurry pH	4.4
Ash content (%)	11.39
Molasses number	450
Iodine number	600
Mean pore radius (Å)	29
Total pore volume (mL/g)	0.93
Apparent density (g/mL)	0.40
Particle density wetted (g/mL)	1.4
Mean particle diameter (mm)	1.0
Effective size (mm)	0.75
Dust (%)	0.196
Uniformity coefficient	1.49
Particle-size distribution, US Sieve Series:	
On 10	0.27%
On 12	3.59%
On 16	44.75%
On 20	31.08%
On 30	16.93%
In pan	3.39%

Carbon Loading

The activated carbon used in the desorption experiments was loaded with Pb and phenol in the column mode. GAC that was washed in distilled water and degassed for 24 hours was placed in a borosilicate glass column using the slurry method and rinsed with 10 BVs of 1 N NaOH over a 3-hour period. The GAC column was then contacted with a solution containing 100 mg/L Pb and 1000 mg/L phenol at a flow rate of 92 mL/min (HLR = 2 gpm/ft^2) until the GAC was exhausted. The mass of carbon used in each column run was approximately 400 g. The average empty bed volume and empty bed contact time (EBCT) were 956 cm^3 and 10.4 minutes, respectively. Two column runs were necessary to produce sufficient carbon for the batch experiments. Normalized breakthrough curves for Pb and phenol are presented for both column runs in Fig. 1. Contaminant surface concentrations (X/M) at exhaustion were calculated using

$$X/M = [V_{inf}C_{inf} - (C_{eff}C_{eff})]/m \quad (1)$$

where X/M = mass of contaminant/mass of carbon, mg/g
V_{inf} = volume of influent wastewater, L
C_{inf} = concentration of contaminant in influent, mg/L
V_{eff} = volume of effluent wastewater collected at GAC exhaustion, L (including samples collected during column run)
C_{eff} = concentration of contaminant in effluent, mg/L
m = mass of carbon in column, g

X/M values for Pb and phenol averaged 12 mg Pb/g and 128 mg phenol/g, respectively. Based on earlier work (4, 5) it was hypothesized that Pb was removed via adsorption and surface precipitation [as $Pb(OH)_{2(s)}$] while phenol removal was attributed to physicochemical adsorption. After removal from the column, the carbon was air dried for 24 hours and stored at 4°C until use in batch experiments.

Desorption Experiments

A summary of experimental conditions is presented in Table 2. There were a total of 108 samples tested (70 at 22°C, 38 at 80°C). Six regeneration schemes were investigated at five different times (0.33, 1, 3, 7 and 21 days). For 22°C, experiments were conducted in triplicate except for the deionized (DI) water controls. For the 80°C experiments, samples were placed in a constant temperature water bath. Four experiments at 80°C were replicated.

Samples were prepared by weighing out 5 or 10 g of exhausted carbon which was then placed in a polyethylene bottle. Regenerant was then

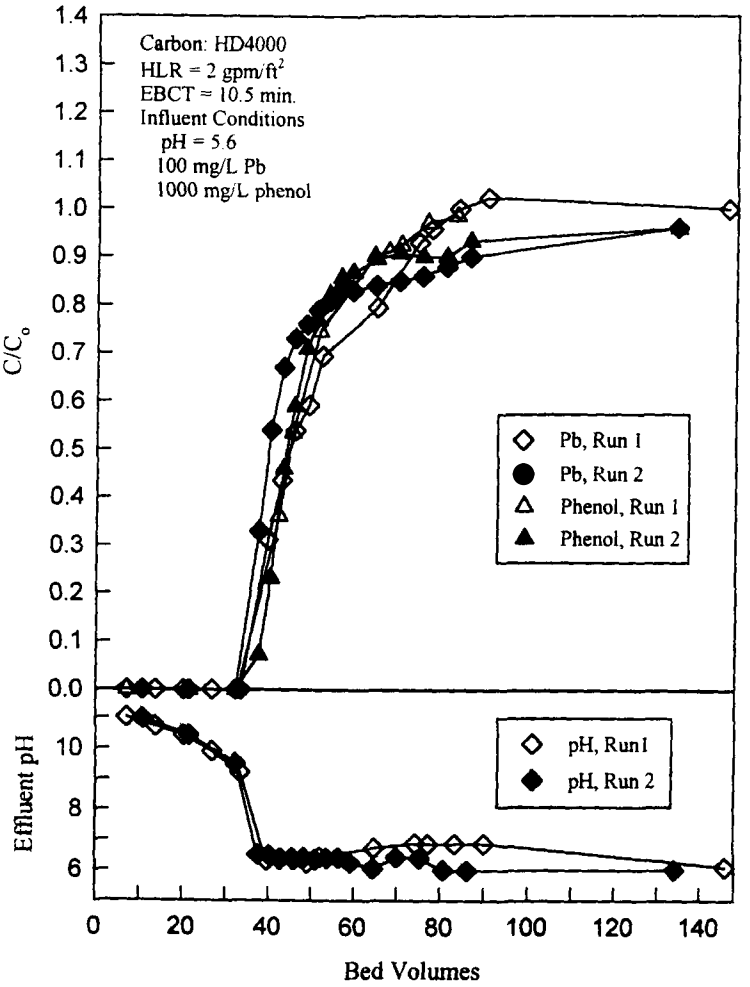

FIG. 1 Normalized breakthrough curves for Pb and phenol for column carbon exhaustions.

added such that the proper R:C was obtained. Samples were placed on a horizontal shaker for the prescribed time period. After shaking, the sample was filtered through a 0.45-μm filter and the filtrate analyzed for pH, Pb, and phenol. Pb and phenol regeneration efficiency was calculated using

$$RE = [X/M_i(m) - (C_{reg}V_{reg})]/[X/M_i(m)] \quad (2)$$

TABLE 2
Summary of Experimental Conditions

Regeneration conditions R:C ratio[a]; Regenerant	Number of tests conducted				
	0.33 d	1 d	3 d	7 d	21 d
		22°C			
5:1; 1 N NaOH	3	3	3	3	3
5:1; 0.1 N NaOH	3	3	3	3	3
10:1; 1 N NaOH	3	3	3	3	3
10:1; 0.1 N NaOH	3	3	3	3	3
5:1; DI water	1	1	1	1	1
10:1; DI water	1	1	1	1	1
		80°C			
5:1; 1 N NaOH	1	1	1	1	3
5:1; 0.1 N NaOH	3	1	1	1	1
10:1; 1 N NaOH	1	1	3	3	1
10:1; 0.1 N NaOH	1	1	1	1	1
5:1; DI water	1	1	1	1	1
10:1; DI water	1	1	1	1	1

[a] Regenerant:carbon mass ratio.

where RE = regeneration efficiency, % (Pb or phenol)
X/M_i = initial contaminant GAC concentration, mg/g
m = mass of GAC used, g
C_{reg} = contaminant concentration in regenerant, mg/L
V_{reg} = volume of regenerant, L

Phenol Degradation and Volatilization Experiment

Deionized water and NaOH solutions containing phenol were heated to 80°C for varying lengths of time to determine if phenol underwent any physical/chemical changes (e.g., degradation, volatilization) due to high pH or temperature. Four solutions were investigated: 1) DI water–1 g/L phenol, 2) DI water–5 g/L phenol, 3) 1.0 N NaOH–1 g/L phenol, and 4) 1.0 N NaOH–5 g/L phenol. Solutions were maintained at 80°C for 0.33, 1, 3, or 7 days.

Analytical Methods

Solutions used during this study were made using reagent-grade chemicals. Pb samples were acidified using concentrated HNO_3 (15 M) such

that the pH was below 2.0. Phenol samples were filtered using a Micronsep 0.45-μm membrane filter and stored at 4°C.

Phenol was analyzed using Standard Methods 5530 D: Direct Photometric Method (1) on a Genesis Model 5 UV-visual spectrophotometer at a wavelength of 500 nm. Pb samples that had a concentration of greater than 0.5 mg/L were analyzed using a flame Perkin-Elmer Model 3100 ZL Atomic Absorption Spectrophotometer at a wavelength of 283.6 nm. Samples that contained less than 0.5 mg/L Pb were measured using a Perkin-Elmer Model 4100-Zeeman graphite furnace at the same wavelength. Analytical recoveries were conducted on at least 10% of the samples at the time of sample measurement. The mean recovery ± one standard deviation was 98.3 ± 5.7% for Pb and 100.1 ± 3.5% for phenol. Based on the excellent recoveries, there did not appear to be a significant analytical interferences.

RESULTS AND DISCUSSION

Experimental Reproducibility

Pb regeneration efficiencies and final pH values versus time for 1.0 N NaOH at 22°C and a R:C of 5:1 are presented in Fig. 2 for three experimental replicates. There is less than a 6% difference in the Pb results from the three duplicates. Phenol regeneration efficiencies and final pH values versus time for 0.1 N NaOH at 22°C and a R:C of 10:1 are presented in Fig. 3 for the replicated samples. The maximum variation was 7%. Results for the other experimental conditions were of similar quality. Because of the excellent experimental reproducibility, one of the three replicates will be presented for each scenario. It will be assumed that the results from the single experiment are representative of process performance. The Pb and phenol sample regeneration results are presented separately in the next two sections.

Pb Regeneration

As mentioned previously, it is hypothesized that Pb was removed via adsorption and surface precipitation mechanisms. Thus, Pb regeneration is accomplished by promoting both desorption and resolubilization. A speciation diagram for Pb in the presence of phenol at representative concentrations is presented in Fig. 4. If solution pH is increased above 12.5, the predominant Pb species is $Pb(OH)_4^{2-}$, resulting in the resolubilization of Pb precipitated as $Pb(OH)_{2(s)}$.

Pb regeneration efficiencies versus time using 1.0 N NaOH and DI water at R:C ratios of 5:1 and 10:1 and temperatures of 22 and 80°C are pre-

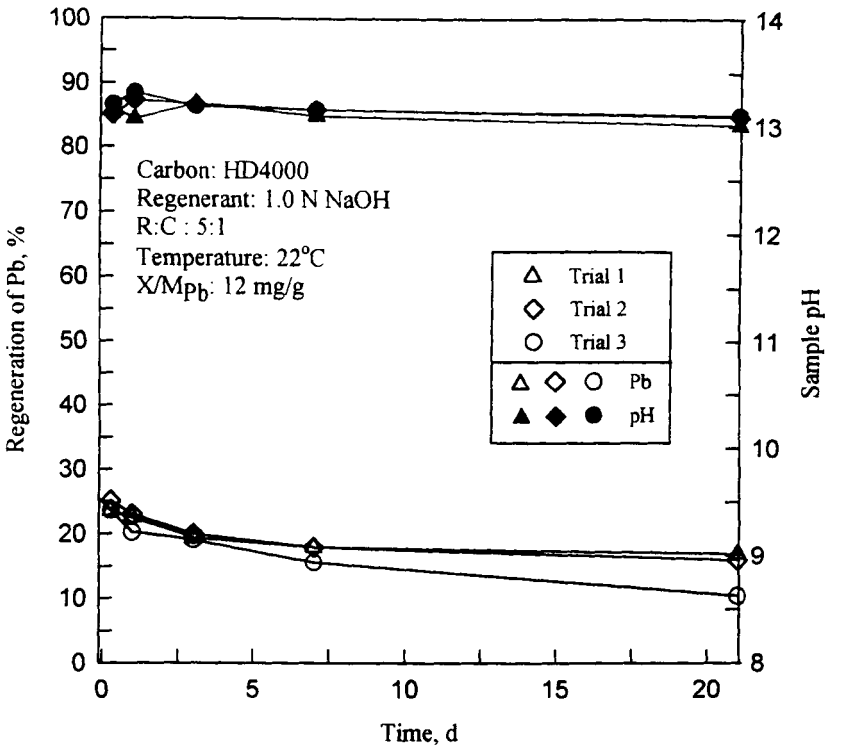

FIG. 2 Pb regeneration efficiencies and final pH values versus time for 1.0 N NaOH at 22°C and a R:C of 5:1.

sented in Fig. 5. Also presented in Fig. 5 are the final pH values of the samples. Increasing the R:C ratio from 5:1 to 10:1 increased the Pb regeneration efficiency by about 20%. The increase in Pb desorption/resolubilization with high R:C can be attributed to 1) a more favorable concentration gradient between the solid and liquid phases (i.e., lower aqueous phase concentration), and 2) more favorable thermodynamic conditions for both Pb desorption and Pb solubilization (i.e., the larger volume of liquid allowed more Pb mass to reside in the aqueous phase for a given aqueous phase concentration).

The difference in Pb regeneration between 22 and 80°C was not significant enough to delinate any trends. Pb desorption/solubilization with DI as the regenerant ranged between about 3 and 8%. The pH of the DI water

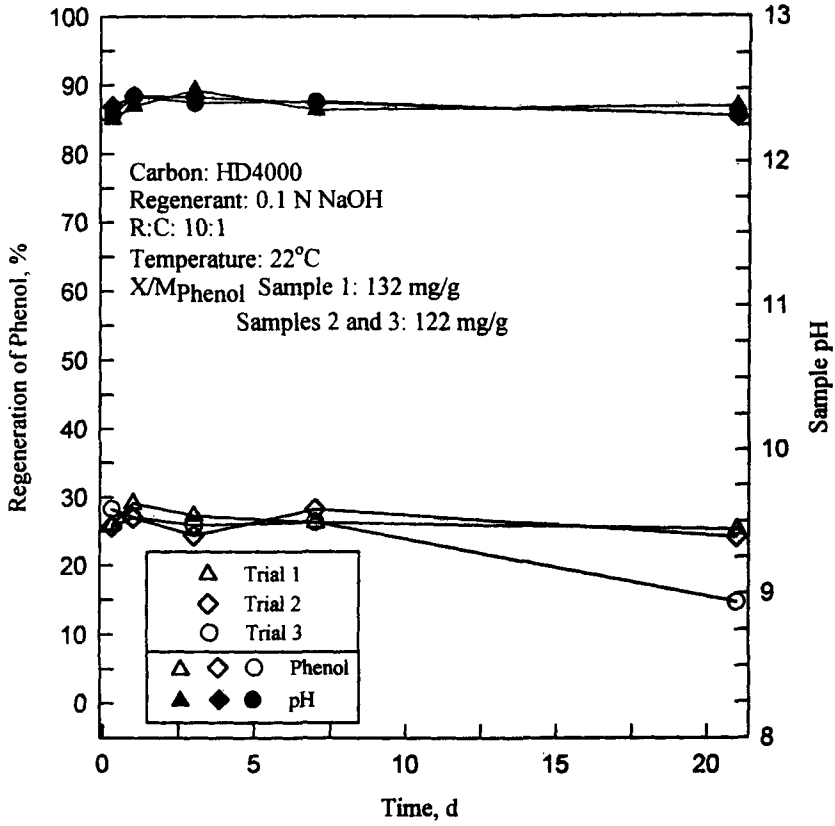

FIG. 3 Phenol regeneration efficiencies and final pH values versus time for 0.1 N NaOH at 22°C and a R:C of 10:1.

samples ranged from 5.5 to 5.8. In this pH range, desorption was not expected because Pb adsorption in this pH region was observed to be significant (4). No matter the pH, a certain amount of surface-precipitated Pb would resolubilize when exposed to a solution containing no Pb. Assuming that $Pb(OH)_{2(s)}$ is the primary Pb solid, the aqueous Pb concentrations, as predicted by thermodynamics, range from 40 to 63 mg/L for the pH range 5.5 to 5.8. In the DI water experiments, Pb solution phase concentrations ranged from 40 to 53 mg/L.

The most interesting observation is the decrease in Pb regeneration efficiency with time. The decrease in Pb regeneration efficiency corre-

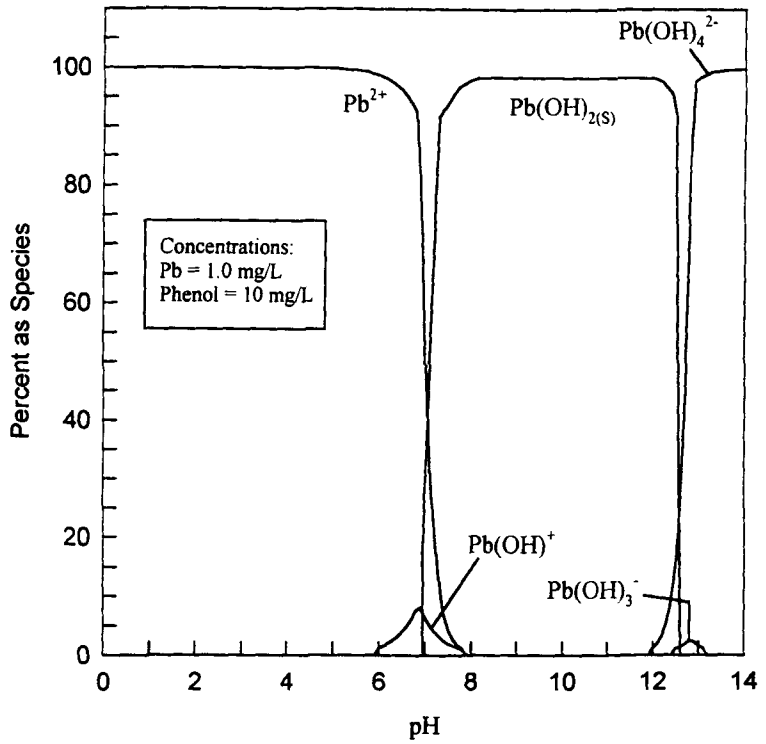

FIG. 4 Speciation of Pb versus pH.

sponds to a decrease in aqueous Pb concentration. Thus, Pb that was desorbed/resolubilized at earlier times was readsorbed or was re-precipitated on the surface. The formation of a more organized, less soluble Pb solid [e.g., crystalline $PbOH_{2(s)}$ compared with amorphous $Pb(OH)_{2(s)}$] may have occurred. It is well known that the formation kinetics of more crystalline solids are slower than those of amorphous solids. Thus, a slower forming solid may have formed at later times from Pb that was desorbed/resolubilized at an earlier time. For example, at pH 13, the solubility of Pb is about 10% lower for crystalline $Pb(OH)_{2(s)}$ compared with amorphous $Pb(OH)_{2(s)}$. If crystalline $Pb(OH)_{2(s)}$ formed at the later times, then the measured aqueous Pb concentration would decrease, resulting in a decrease in regeneration efficiency. Another possibility is that the initial loading of the carbon was incomplete (i.e., did not reach equilib-

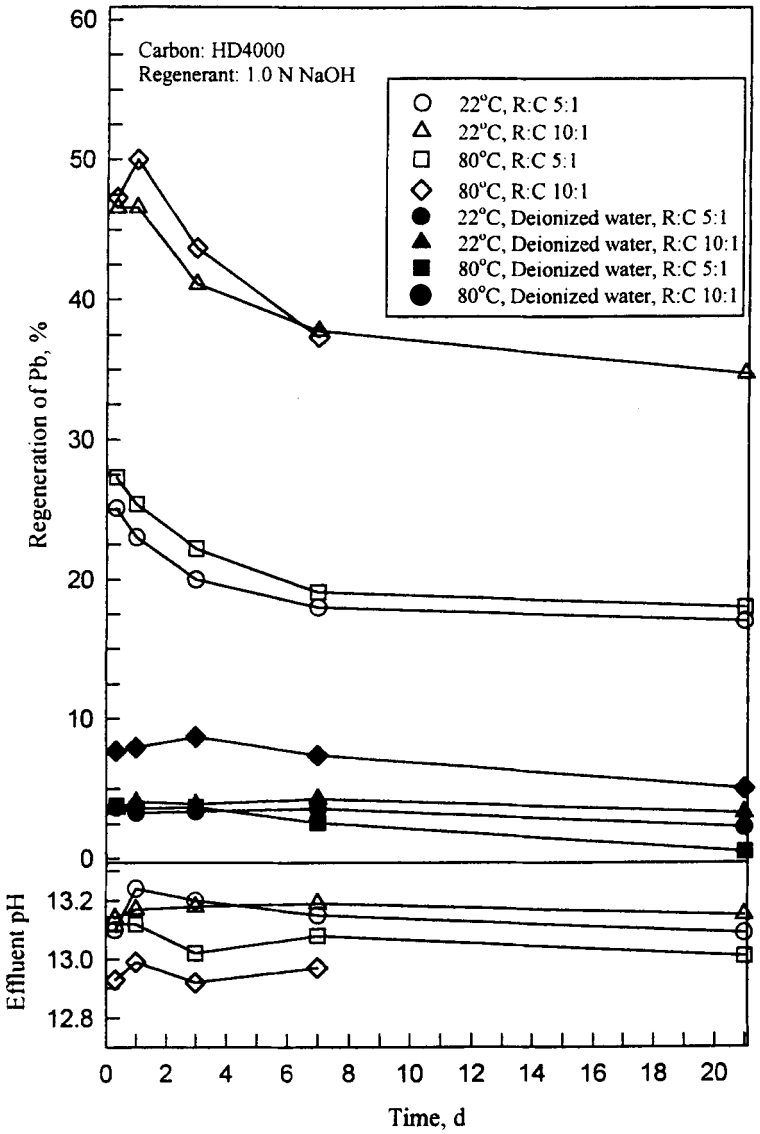

FIG. 5 Pb regeneration efficiencies versus time using 1.0 N NaOH and DI water at R:C ratios of 5:1 and 10:1 and temperatures of 22 and 80°C.

rium). If this is the case, Pb in solution during regeneration would have decreased as time increased as the GAC readsorbed the Pb present in the regenerant.

Pb regeneration efficiencies and pH versus time for 0.1 N NaOH and DI water are presented in Fig. 6. Pb regeneration efficiencies using 0.1 N NaOH were markedly lower than those observed at 1.0 N NaOH, most likely due to the system pH. For 0.1 N NaOH, the pH was between 11.3 and 12.5, about one pH unit lower than the 1 N NaOH. In the pH range

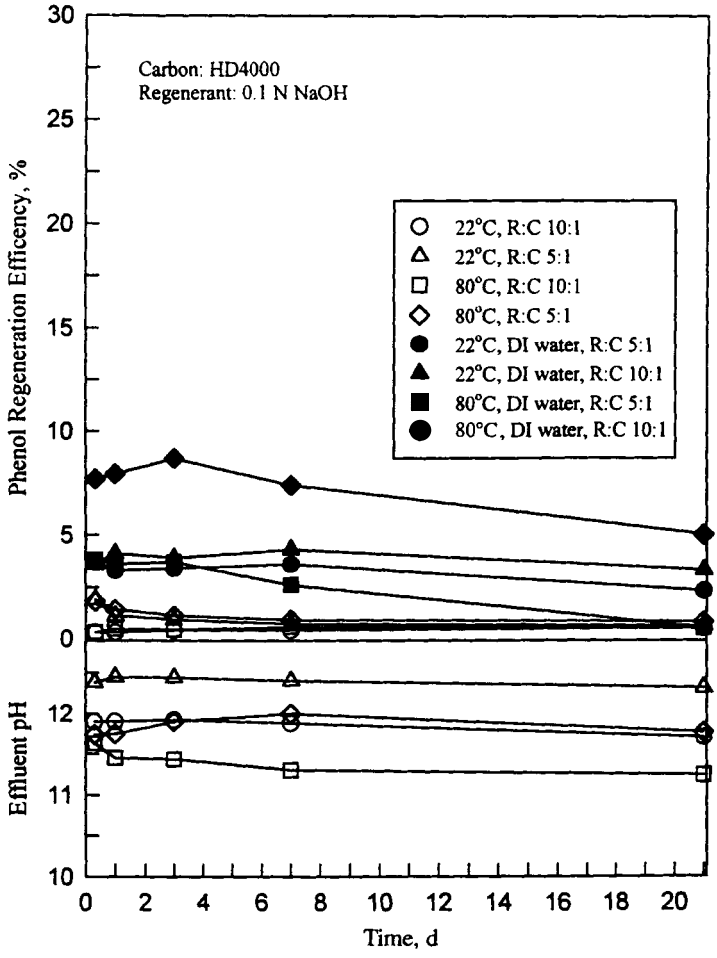

FIG. 6 Pb regeneration efficiencies and pH versus time for 0.1 N NaOH and DI water.

11 to 12.5, $Pb(OH)_{2(s)}$ solubilization is insignificant. Thus, a decrease in regeneration efficiency (decrease in aqueous Pb concentration) was expected. Similar results were reported by Reed et al. (5). In addition, the ionic strength was much lower for 0.1 N NaOH compared with the 1 N system. Several authors reported that heavy metal removal by activated carbon decreases with increasing ionic strength (3, 4), thus regeneration efficiencies should be higher at higher ionic strengths.

Phenol Degradation/Volatilization

Phenol transformation (i.e., losses in sample) can be caused by 1) volatilization, 2) biological degradation, and 3) chemical degradation (aided by higher temperatures). Degradation/volatilization experiments were conducted to estimate how phenol transformation would affect the regeneration efficiency of phenol-loaded GAC. If a significant amount of phenol was degraded/volatilized, then the apparent (or calculated) regeneration efficiency would be less than the actual efficiency. Normalized phenol concentrations versus time for the four experimental solutions are presented in Fig. 7. In 7 days the phenol concentration in both DI water and NaOH decreased an average of 9 and 8%, respectively. The loss of phenol was greater for DI water than for the NaOH solution. Phenol in the DI water (pH 5.5 to 5.8) exists as C_6H_5OH while in NaOH (pH > 11) phenol exists as $C_6H_5O^-$. Only C_6H_5OH is volatile, thus the larger phenol loss in DI water could be due to volatilization. There may also have been less biological activity in the NaOH samples compared with the DI water because of the high pH values of the NaOH solutions. However, no biomass was observed in any of the solutions. Chemical destruction would most likely have occurred in the caustic solutions. Given that the concentration of phenol in the NaOH solutions decreased an average of only 8%, the importance of chemical destruction appears to be minimal. Regardless of the exact transformation mechanism(s), the amount of phenol that was lost by degradation/volatilization was minimal and the apparent phenol regeneration efficiencies calculated in the GAC experiments can be considered to be an accurate estimate of the actual efficiencies.

Phenol Regeneration

As mentioned previously, it is hypothesized that the activated carbon removal mechanism for phenol is physiochemical adsorption. Desorption of phenol can be caused by the formation of the less adsorbable $C_6H_5O^-$.

Phenol regeneration efficiencies versus time are presented in Fig. 8 for 1.0 N NaOH and DI water and Fig. 9 for 0.1 N NaOH and DI water. For both temperatures and regenerant solutions, regeneration efficiencies

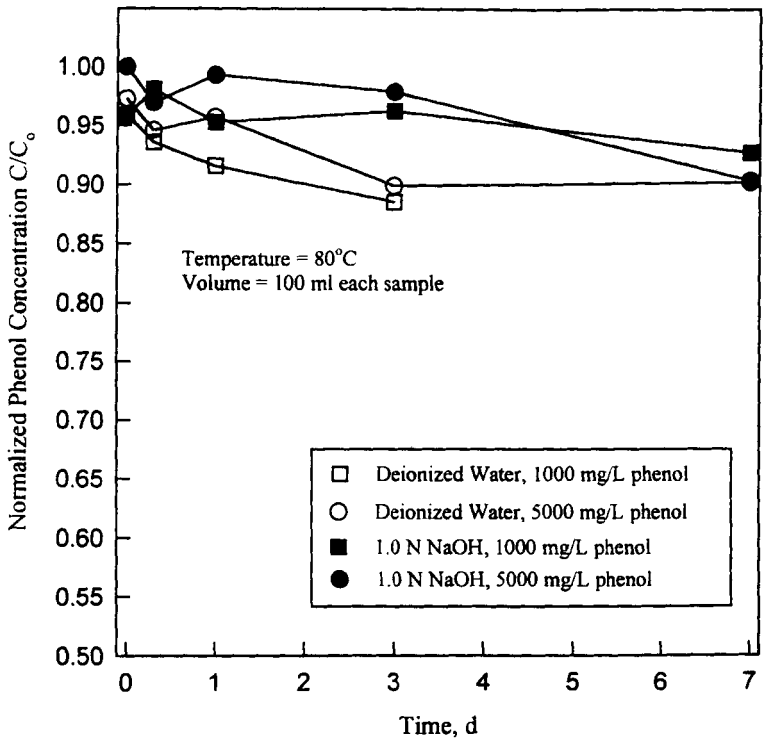

FIG. 7 Normalized phenol concentrations versus time for four experimental solutions.

were higher for the 10:1 R:C ratio compared with an R:C of 5:1. Reasons for this behavior were discussed earlier for Pb. The effect of R:C ratio was less apparent at 80°C. In general, phenol regeneration efficiencies were lower at 80°C. The difference in regeneration efficiencies (not necessarily regeneration effectiveness) at the two temperatures can be attributed to 1) phenol losses via degradation/volatilization and 2) the delay in phenol deprotonation ($C_6H_5OH \rightarrow C_6H_5O^-$) with respect to pH. For the later scenario, the pK_a for phenol increases by 1.5 log units (9.9 to 11.4) as the temperature is increased from 22 to 80°C. Thus, for a given pH, more of the phenol exists as the adsorbable C_6H_5OH.

Phenol regeneration efficiencies for 1 N NaOH were higher by about 10 to 20% compared to 0.1 N NaOH experiments. For the stronger caustic solution there was more $C_6H_5O^-$ present because of the higher pH. In addition, the phenol pK_a decreases with increasing ionic strength (i.e.,

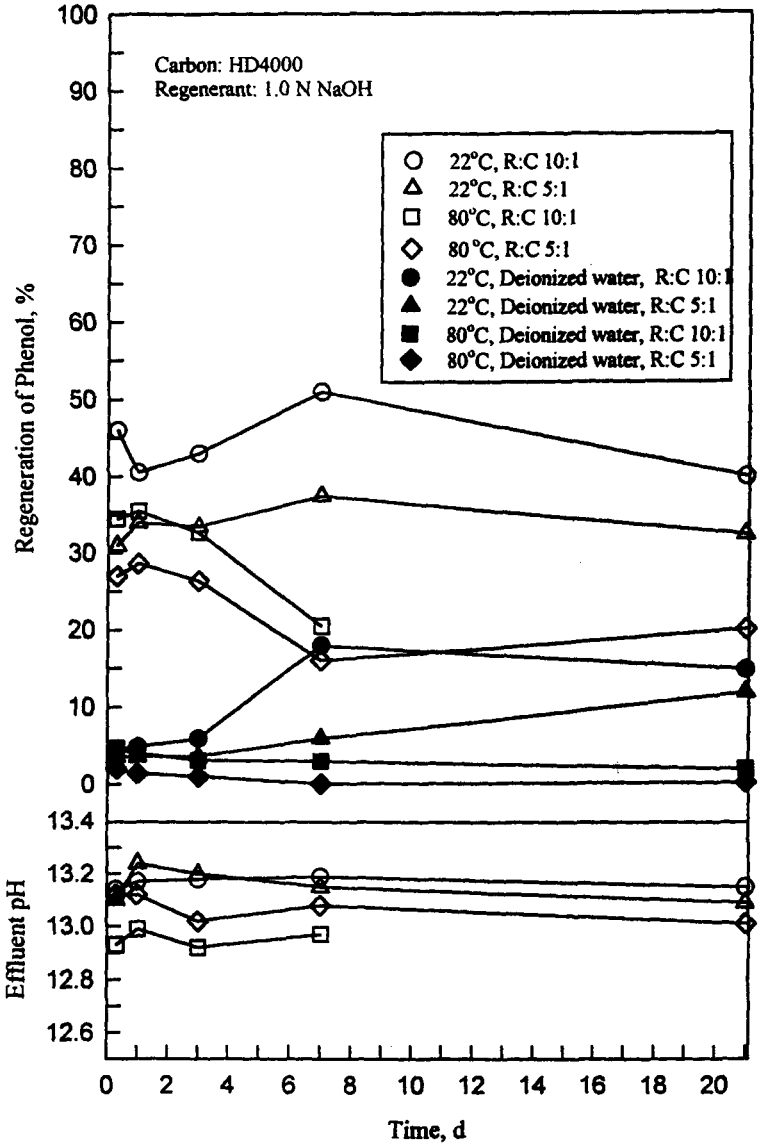

FIG. 8 Phenol regeneration efficiency versus time for 1.0 N NaOH.

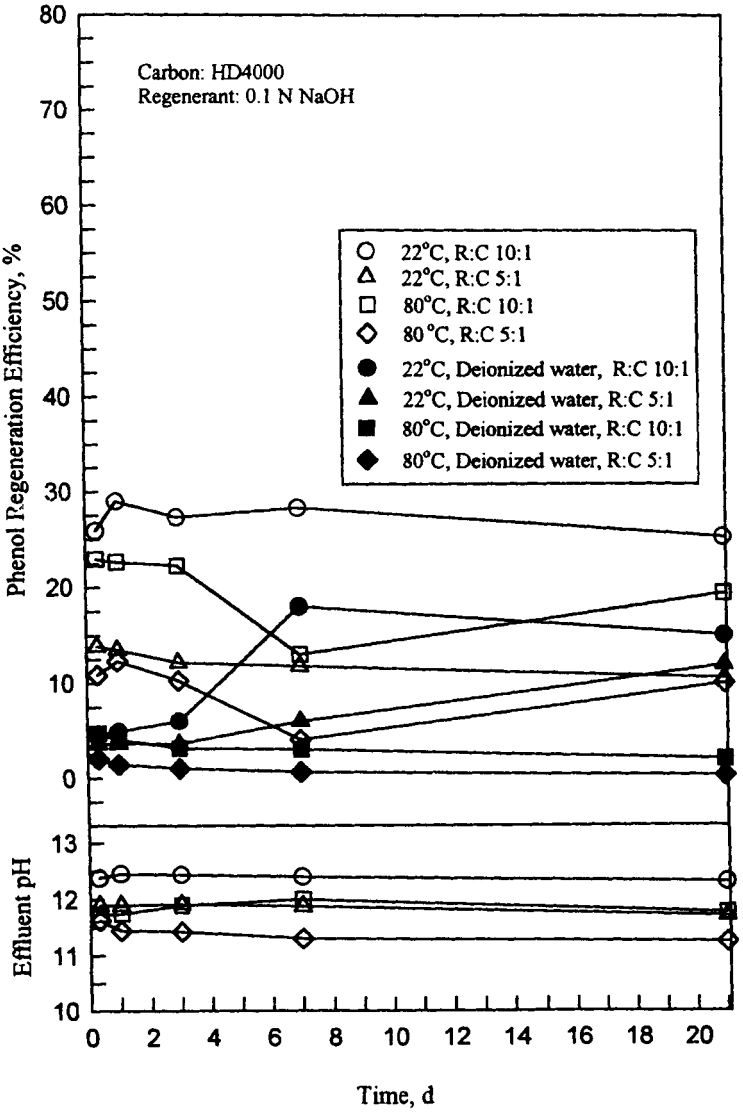

FIG. 9 Phenol regeneration efficiencies versus time for 0.1 N NaOH.

TABLE 3
Pb and Phenol Regeneration Efficiencies at 8 Hours

Regeneration conditions R:C ratio[a]; regenerant	Pb		Phenol	
	22°C	80°C	22°C	80°C
5:1; 1 N NaOH	24	27	26	27
5:1; 0.1 N NaOH	0.3	0.3	14	11
10:1; 1 N NaOH	47	47	42	35
10:1; 0.1 N NaOH	2	2	26	23
5:1; DI water	4	4	4	2
10:1; DI water	4	8	5	5

[a] Regenerant:carbon mass ratio.

phenol pC–pH diagram shifts toward lower pH values). For a given pH, more of the phenol exists as $C_6H_5O^-$.

For 22°C, phenol regenerations did not change significantly with time. However, for 80°C experiments the regeneration efficiency decreased with time (phenol in the aqueous phase decreased) for the caustic regenerant but not for DI water. For the caustic solution, phenol desorbed from the surface, and a portion may have been degraded/volatilized. For the 80°C DI water experiments, there was little desorption of phenol, thus there was less of an opportunity for degradation/volatilization.

Comparison of Regeneration Efficiencies

In Table 3, Pb and phenol regeneration efficiencies at 0.33 days are presented for the study regenerant solutions and temperatures. A time of 8 hours was selected because for many regeneration conditions the efficiencies were highest at this time and in an actual treatment system relatively short regeneration times would be used. Temperature had little effect on both Pb and phenol regeneration efficiencies, while increasing the R:C ratio increased regeneration. The 0.1 N NaOH was ineffective for Pb. Increasing the strength of the caustic solution from 0.1 to 1 N improved phenol desorption. The optimum conditions for Pb and phenol regeneration were 22°C, R:C = 10:1, and 1 N NaOH.

CONCLUSIONS

Based on batch regeneration experiments, NaOH is a viable regenerant for GAC loaded with Pb and phenol. 1.0 N NaOH removed a higher percentage of Pb and phenol from the GAC than did 0.1 N NaOH. More

contaminant was removed at an R:C of 10:1 than at an R:C of 5:1 for both Pb and phenol. For Pb, maximum regenerative efficiencies occurred within the first 24 hours, and usually within the first 8 hours. The decrease in regenerative efficiency with increasing time may be a result of the formation over time of more stable Pb solids (i.e., amorphous to crystalline), or from a continued adsorption of contaminants by GAC which has not yet come to equilibrium. For phenol, the maximum regenerative efficiencies occurred within the first 3 days, and did not appreciably decrease with time. In an actual treatment system, higher regeneration efficiencies would be expected because the exhausted carbon would undergo serial regenerations or be regenerated in the continuous mode.

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