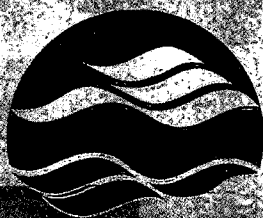


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# The Evaluation of Polymeric Absorbents For In-Situ Groundwater Remediation.

BY:

S. Lesage, M. Venhuis, K. Millar, A. Crowe

NWRI Contribution No. 98-244

## MANAGEMENT PERSPECTIVE

**Title:** The Evaluation Of Polymeric Absorbents For *In-Situ* Groundwater Remediation.

National Water Research Institute, Environment Canada.

**Author(s):** S. Lesage, M. Venhuis, K.R. Millar and A.S. Crowe

**NWRI Publication #:** 98-244

**Citation:** Journal article to be submitted to the Water Quality Journal of Canada.

**EC Priority/Issue:** This work was conducted at NWRI. It was funded by Environment Canada under the Clean Environment (Toxic) Issue.

**Current Status:** This work is now complete. This was a preliminary investigation to find out whether these new products that are currently manufactured to cleanup spills had potential for the remediation of groundwater. The polymeric beads from two different manufacturers were compared to activated carbon for their capacity to remove dissolved contaminants. While they are excellent to clean solvents and gasoline spills, they are inferior to carbon for dissolved compounds.

**Next Steps:** No further work planned.

## Evaluation of Polyalkastylene Absorbent Beads for the Remediation of PCE in Groundwater

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Passive groundwater remediation technologies are increasingly considered because they require very little long-term maintenance and are thus more cost effective than active systems such as pump-and-treat, which require the continuous upkeep of a groundwater pumping system and of an above-ground water treatment plant. Barriers are being installed in the ground to either prevent the flow of contaminated water from one property to another, or to remove the contaminants from the water as it passes through a porous reactive barrier. Absorbents which irreversibly incorporate spilled solvents into their structure have been developed to clean surface spills. This project was aimed at evaluating the performance of the absorbents with dissolved organic contaminants. It was found that while they did sorb aqueous phase compounds, it was through adsorption, not absorption and that it was therefore reversible. As adsorbents, the polymers marketed as Imbiber Beads® and Expandabeads® were not as effective as activated carbon.

**Key words:** imbiberbeads, polyalkastylene beads, absorption, PCE, activated carbon

### Introduction

Although over 30% of the Canadian population depends on groundwater as their source of drinking water, contamination of this valuable resource occurs all too frequently due to accidental spills, leakages or improper disposal practices. Of the many contaminants found in groundwater, organic compounds have been recognized as the contaminant of greatest concern, with tetrachloroethylene (PCE) being one of the most common (Pankow and Cherry 1996). PCE is a volatile chlorinated hydrocarbon widely used in industry as a degreasing agent and as a dry-cleaning solvent.

Because PCE is denser than water and poorly soluble, it is classified as a DNAPL (dense non-aqueous phase liquid). A DNAPL will tend to migrate downward by gravity through the vadose zone, past the water table and continue downward through the unsaturated zone until it encounters a zone of low permeability. As it migrates downward within the saturated zone, some of the free product will remain throughout the soil as a residual. This residual product will dissolve into the groundwater and migrate in the direction of groundwater flow creating a plume of contaminated groundwater much larger than the source of DNAPL. It

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is this plume of dissolved phase PCE that can pose the greatest concern to groundwater resources.

Many techniques have been developed to remediate groundwater containing dissolved phase contaminants (Domenico and Schwartz 1997). The most common one, called "pump-and-treat", consists of pumping contaminated groundwater to the surface and treating it using methods such as air stripping, UV-enhanced oxidation or biological degradation. Adsorption on charcoal, most often used as a final step, has been found effective and reliable in the removal of low solubility organic compounds, some metals, and inorganic species (Barcelona et al. 1988).

It is also possible to use in-situ treatment techniques to remediate contaminated groundwater. In-situ techniques are increasingly gaining acceptance because the long-term maintenance costs of pump-and-treat are very high. One in-situ treatment technique commonly used to treat migrating contaminant plumes is a permeable treatment barrier (Barcelona et al. 1988). Permeable treatment beds involve the excavation of trenches which are filled with a reactive permeable medium, such as activated carbon or limestone. Dissolved phase contaminants in the groundwater flowing through this wall will either react with, or be sorbed onto, the bed material. Thus, groundwater exiting the reactive barrier will contain essentially no contaminants. One advantage of this process is its low maintenance cost. However, disadvantages include the short life of the reactants and sorbents, the possibility of plugging of the barrier, which could cause channeling of the contaminants, and the eventual desorption of the contaminants from the sorbent.

Most sorbents have been developed for above-ground application for the cleanup of spills in industry. There are two types of sorbents: adsorbents and absorbents. Absorption is defined as "a process where the material taken up is distributed throughout the body of the absorbing material", whereas adsorption is "a process where the material taken up is distributed over the surface of the adsorbing material" (ASTM 1995). In the case of adsorption, the contaminant can be released again by heating or washing with a surfactant or solvent. Most sorbents on the market, including activated carbon, are adsorbents. An absorbent, on the other hand, absorbs the contaminant within its polymer structure such that it cannot be removed by physical means. Two absorbents that are currently marketed for spill cleanups are Expandabeads® (Big 'O' Inc., Exeter Ont.) and Imbibibeads® (Imbibitive Technologies Corporation, St. Catharines, Ont.). Both products are alkastyrene copolymer beads and are virtually identical, although Expandabeads® is sold with varying amounts of a wick material to prevent the beads from sticking to each other. These materials have the capability of sorbing large quantities of organic solvents and permanently retaining them within their polymer structure, thus removing the possibility of desorption. These products have been tested and are currently being sold to clean up solvent spills on both land and water. However, their use with water contaminated with dissolved organic compounds has not been examined.

The purpose of this project was to determine the feasibility of using the polymer beads to sorb organic contaminants in the subsurface, either as free phase or dissolved in groundwater. When a solvent contacts these polymeric beads, it causes a swelling of the beads. The advantage of this material is its potential to form a self-sealing barrier that could prevent further migration of DNAPL. The contaminants that are absorbed would also not be re-released to the environment, resulting in a very long life expectancy for the wall.

The project was divided into two parts. The first was to estimate the capability of the polymer to absorb PCE, using batch tests. The second half of the project was to examine the beads as an in-situ barrier for dissolved PCE, in a column setting, which would be more representative of a flowing groundwater system. The efficiency of sorption of the polymers was compared to that of granular activated carbon.

### Sorption Isotherms

Sorption is a term that describes the processes occurring at the solid-liquid interface, and includes adsorption, absorption, ion exchange and partitioning. A sorption isotherm is a plot of the concentration of the compound sorbed (the sorbate) onto a solid (the sorbent) versus the concentration remaining in solution, at a given temperature. At dilute concentrations, chlorinated solvents often display a linear relationship with an intercept of zero. The slope of a linear sorption isotherm can be used to determine a partitioning coefficient,  $K_d$ , for a given compound:

$$K_d = C_s / C_w \quad (1)$$

where  $C_s$  is the concentration of the compound sorbed (mass sorbate/mass solid) and  $C_w$  is the concentration of the compound remaining in solution (mass solute/volume of water).

### Measurement of Sorption

Two types of methods are commonly used in a laboratory to measure the sorption of organic compounds: batch tests and column tests. Batch tests measure the  $K_d$  for a discrete sample and, because they allow maximum contact between solids and solution, they represent the maximum amount of sorption possible. Column tests are more representative of the natural field settings where the amount of sorption depends on the volume of liquid contacting the sorbent and on contact time, which is dependent on flow. An aqueous solution of the contaminant is passed through the sorbent material packed in a column. Conservative tracers are initially used to measure the pore volume (also called void volume) of the column. The breakthrough curve of the sorbate is compared to that of the tracer and a retardation factor  $R_f$  for the sorbate is determined. For a linear reversible sorbing solute on a homogeneous porous media, the  $K_d$  can be determined by the use of equation 2:

$$R_f = 1 + (\rho_b K_d) / \theta_w \quad (2)$$

where  $\rho_b$  is the bulk density of the solid and  $\theta_w$  is the water filled porosity.

### Effects on Sorption

The sorption of a compound depends mostly on its chemical structure, but can also be influenced by the presence of other compounds in the aqueous phase. In general, any mechanism that increases the solubility of a compound will decrease sorption and vice versa. Geochemical parameters that influence sorption include pH, temperature and ionic strength. Solubility generally increases with temperature and decreases with increasing ionic strength. The solubility of halogenated non-ionizable organic compounds is generally not strongly influenced by these geochemical factors, as was shown for trichloroethene (Pavlosthathis and Jaglal 1991). The presence of water-miscible co-solvents and of surfactants have a much stronger influence on the sorption of organic compounds.

The type of sorbent also has a large effect on sorption. For example, while examining the potential for different construction material for groundwater sampling, Reynolds et al. (1990) found that latex rubber had the highest affinity for bromoform, followed by low-density polyethylene. Teflon was the least sorptive polymer. At the other end of the spectrum, many polymeric material such as octadecylsilylated silica, which are designed to sorb organic compounds from water and are used in analytical chemistry, have a strong affinity for tetrachloroethylene (Lesage et al. 1996).

There is a direct relationship between the absorption rate and the surface area of the sorbent in contact with the sorbate. The larger the surface area, the higher the sorption rate. Absorption is also dependent on the contact time between the sorbent and the sorbate. Absorption is a slower process than adsorption because penetration into the bead by diffusion takes more time than adsorption onto a surface (Appelo and Postma 1993).

The chemical properties of the contaminant also affect absorption rate. Non-polar, low viscosity organic chemicals, such as gasoline, are absorbed the fastest. More viscous chemicals require greater time to be absorbed. As organic compounds are absorbed, they become a minority part of the bead, increasing its affinity for the compound being absorbed. As low viscosity compounds, such as toluene, benzene and chlorinated solvents, are sorbed, the affinity of the bead also becomes greater for more viscous compounds having similar hydrophobicity, increasing the absorption rate for these compounds. The beads have the capacity to absorb up to 27 times their volume (Big 'O' Inc., [b], no date). As organic compounds are imbibed within the polymer structure, the beads swell and become sticky, transforming to a gel-like solid.

## Materials and Methods

### Batch Tests

A series of batch tests were conducted to determine the capability of Expandabeads® to absorb PCE. Sample sets were prepared as 40-mL solutions in 50-mL serum bottles containing Expandabeads® (0.5, 0.7, 0.9, 1.25 g), activated carbon (0.1, 0.2, 0.25 g) or Expandabeads® with wick (0.5 g of 100%, 70%, 35% beads). The exact composition of the wick was not disclosed by the manufacturer, but was a white solid flaky material of waxy plastic appearance, up to 0.5 cm in diameter and approximately 1 mm in thickness, with uneven edges. Solutions were prepared in duplicate containing 0.1 mg/L and 1.0 mg/L PCE, with the exception of the wick mixtures, which were done in duplicate with only 1.0 mg/L PCE. Vials were sealed with Teflon<sup>®</sup>-lined butyl rubber stoppers and aluminum crimp caps.

PCE was measured by sampling the headspace of the serum bottle with a 50- $\mu$ L gas-tight syringe and injecting into a Photovac 10S+ portable gas chromatograph (Markham, Ont.) equipped with a 10.6 eV lamp. The column was a CPSIL-5 capillary column. Analyses were isothermal at 50°C using air ultra zero as the carrier gas. PCE standards were made in water in similar serum bottles as the samples.

Comparison of Expandabeads® and Imbibebeads® was also made. Samples consisted of either Expandabeads® (0.5 g) or Imbibebeads® (0.5g), 40 mL of water and 1.0 mg/L PCE. Desorption of PCE from the beads was evaluated using the vials containing the wicking material and carbon by the addition of methanol (2 mL), surfactant (Tween 40; 3 mL, 1% wt/wt), heat (water bath; 30 min. at 80°C), and change of pH by the addition of HCl (0.1 M, 2 mL) or NaOH (0.1 M, 4 mL).

### Column Tests

Experiments were conducted in a glass column 4.8 cm (i.d.)  $\times$  30 cm (in length) filled with size 20/30 glass beads. The columns were slurry packed. Three separate column experiments were conducted where a layer, consisting of either 1 or 10% by weight of Expandabeads® in glass beads or 10% by weight of activated carbon in glass beads, was included in the middle of the column, simulating a contaminant-intercepting barrier in a groundwater flowing system.

To determine the pore volume of the column and to ensure no channeling was present, a tracer test was then run for each column using a 2-mL slug of 1% sodium chloride. A simple flow cell was set up to accommodate a chloride electrode and provide continuous monitoring of the effluent (Fig. 1). The pore volume was determined from the breakthrough curve of the chart recorder trace.

After the tracer test, the column was washed with several pore volumes of water to remove the sodium chloride. A solution of 100 mg/L PCE was pumped continuously through the columns at a rate of 0.28 mL/min, giving residence times of 11-12 hours. Solutions were kept in a Tedlar®

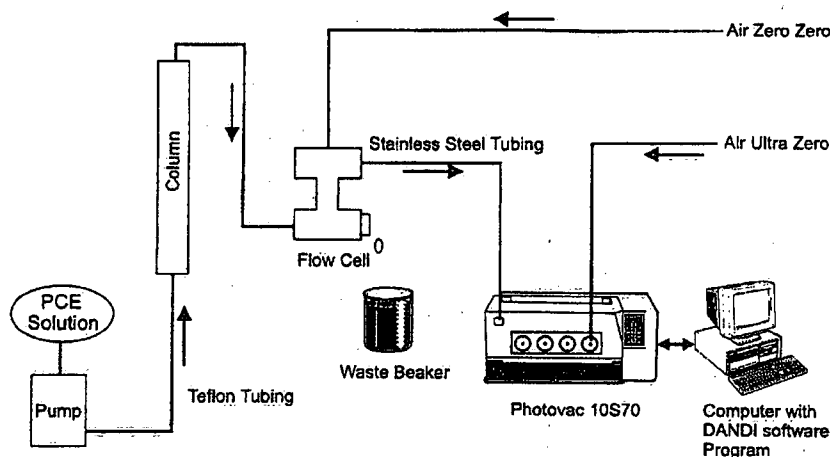


Fig. 1. Schematic representation of the experimental setup for the flowthrough sorption experiment.

bag with no headspace. The column effluent was pumped through a flow cell (Lesage and Brown 1994) where it could be measured for PCE using a Photovac 10S70 portable gas chromatograph (Markham, Ont.) equipped with a 10.6 eV lamp. Air zero was used as the make-up gas to the flow cell at a rate of 10 mL/min. Analyses were isothermal at 40°C using air ultra zero as the carrier gas flowing at 15 mL/min. Headspace samples (1.4 mL) were automatically taken at 2-hour intervals. Data acquisition was done using the Photovac PC Dandi™ software program (Photovac Inc., Markham Ont.) in a laptop computer interfaced to the gas chromatograph. Calibration of the flow cell was done by pumping the PCE solution through the flow cell for at least 2 hours and taking samples every 10 minutes. The average of 10 analyses was used to calibrate the GC.

After several pore volumes of PCE were pumped through the column, the PCE solution was replaced with water to study the desorption of PCE from the beads.

## Results

### Batch Tests

Figures 2a and b show the sorption of PCE in samples containing increasing amounts of beads. With initial concentrations of 0.1 and 1.0 mg/L PCE, all amounts of beads were found to sorb in excess of 80% of the PCE within the first 100 hours. At the highest concentration of PCE, sorption was rapid with 70% removal in 5 hours. However, after 30 hours, the absorption seemed to cease with possible desorption in the samples containing 0.5 and 0.7 g of beads. The 0.1-mg/L PCE solutions showed only



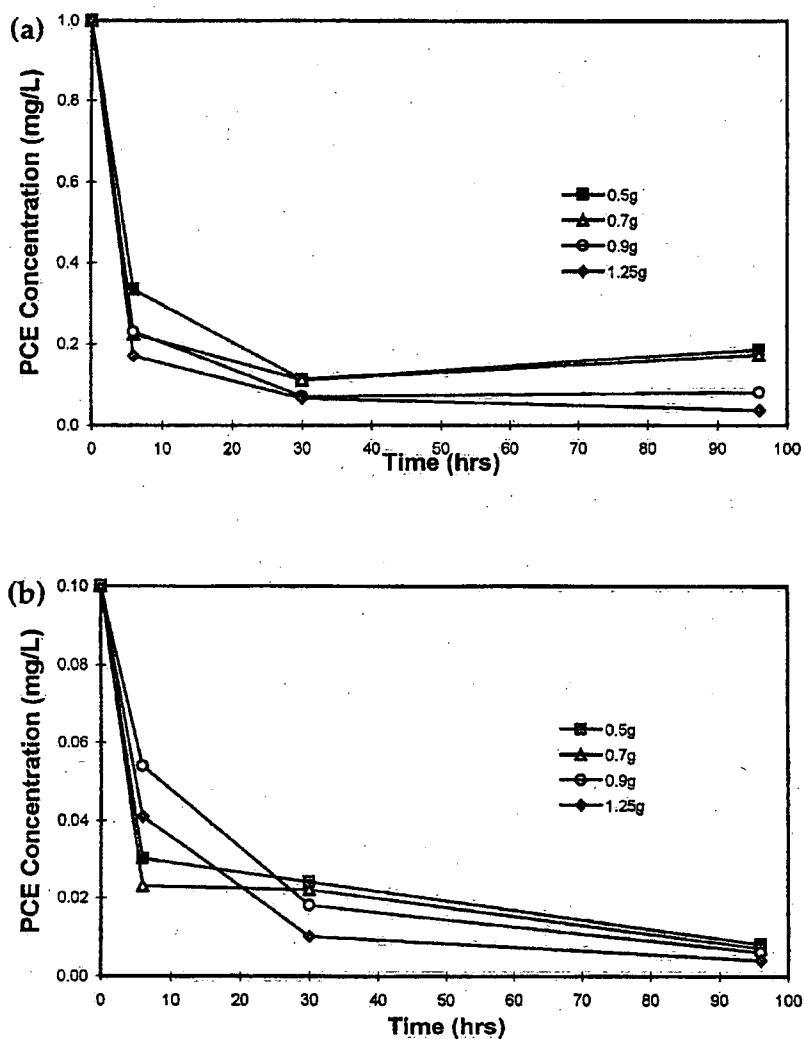


Fig. 2. Batch test sorption of PCE on Expandabeads®. Initial concentrations: (a) 1.0 mg/L PCE; (b) 0.1 mg/L PCE.

a 40–80% removal in the first 5 hours. The amount sorbed was not correlated significantly to the amount of beads present, probably because they were in excess at all concentrations. Figures 3a and b show the absorption of 1.0 mg/L and 0.1 mg/L PCE on increasing amounts of carbon. Initial sorption in excess of 98% was observed at all concentrations of carbon in samples initially containing 1.0 mg/L PCE. In the sample set containing 0.1 mg/L PCE initially, sorption averaged 70% within the first 7 hours. By 90 hours, PCE was below detection limit in all samples.

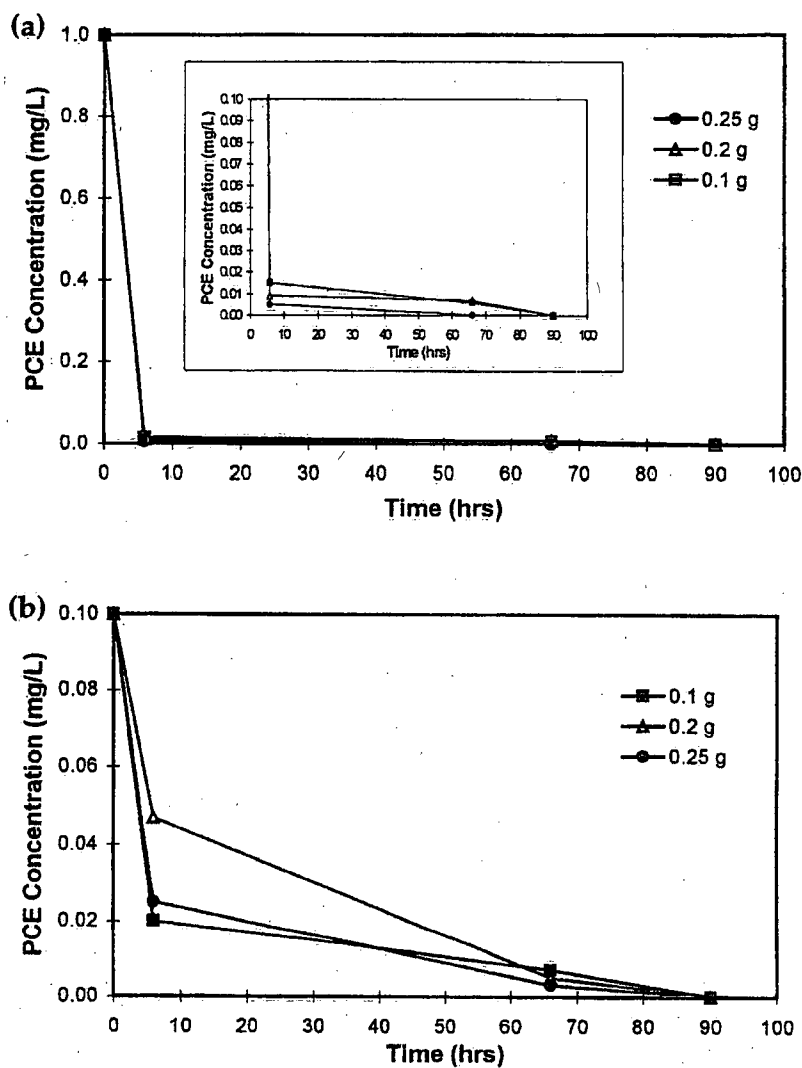


Fig. 3. Batch test sorption on activated carbon. Initial concentrations: (a) 1.0 mg/L PCE (inset: expansion of y scale, 0–0.1 mg/L); (b) 0.1 mg/L PCE.

Sorption of PCE using Expandabeads® was also examined with different amounts of wicking material (Fig. 4). Initially sorption was enhanced with increasing amounts of wicking material. However, by 90 hours, there was no significant difference between treatments. The comparison between Expandabeads® and Imbibebeads® is shown on Fig. 5. Initially the Imbibebeads® sorbed more than the Expandabeads®, but there was no difference by 70 hours. All attempts to desorb PCE from the beads and

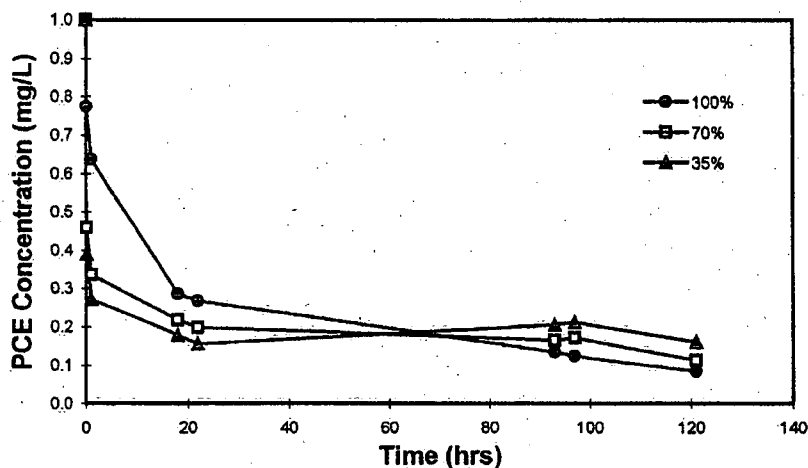


Fig. 4. Batch test sorption results of 1.0 mg/L PCE on different Expandabeads® and wick combinations.

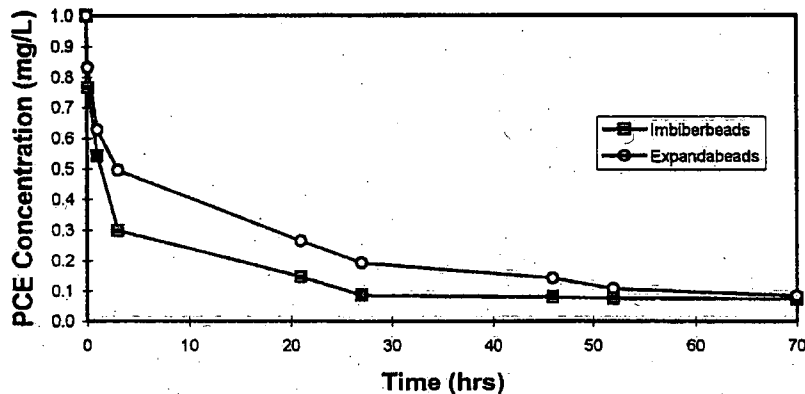


Fig. 5. Comparison between Imbibebeads® and Expandabeads® in batch sorption tests.

carbon by means of pH change, heat, methanol and surfactant additions were ineffective.

#### Column Tests

Figure 6 shows the breakthrough curve for the first column test performed with a layer containing 1% Expandabeads® in glass beads. Less than 2 pore volumes were required before PCE breakthrough. After 7 pore volumes, periodic spikes were observed and continued through out the

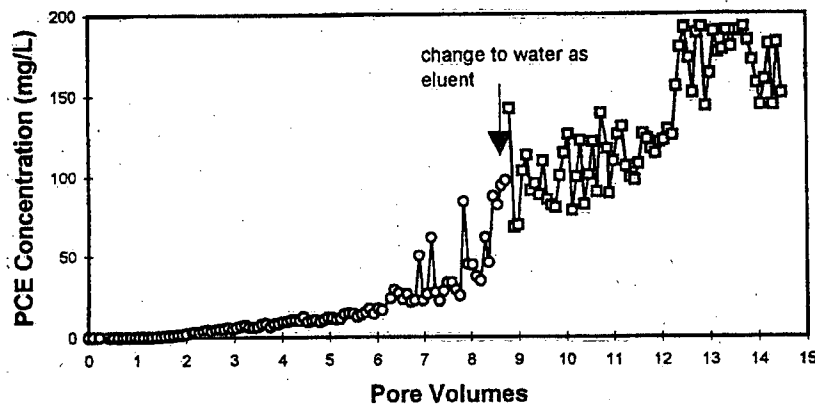


Fig. 6. Column test results for a 1% Expandabeads®/glass mixture. A saturated solution of PCE was pumped through the column for nine pore volumes, followed by a water wash to examine desorption.

remainder of the test. After 9 pore volumes, the pumping of PCE solution was stopped and replaced with water. After this point, the concentrations of PCE continued to increase.

The breakthrough curve for the 10% Expandabeads® mixture column is shown in Fig. 7. Breakthrough of PCE occurred in less than 10 pore volumes. As the PCE concentration rose, the scatter of results increased.

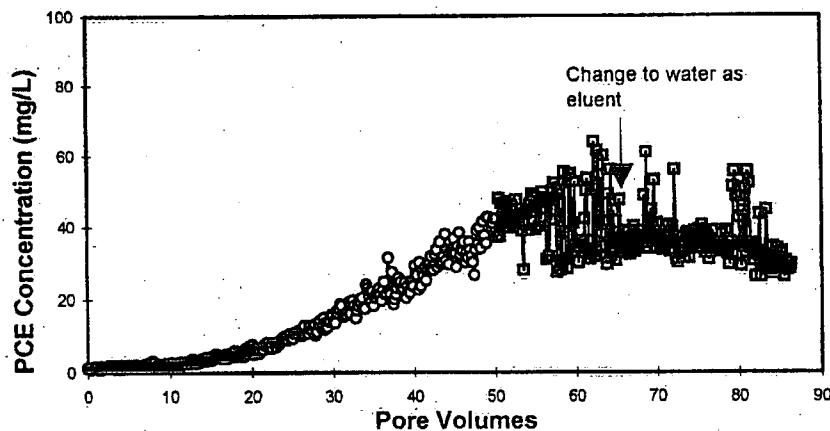


Fig. 7. Column test results for a 10% Expandabeads®/glass mixture. A saturated solution of PCE was pumped through the column for 50 pore volumes, followed by a water wash to examine desorption.

After 50 pore volumes, the eluent was changed to water and it took more than 20 pore volumes before a decrease in PCE concentrations was observed.

In the column containing a 10% activated carbon layer, no PCE breakthrough was observed even after 60 pore volumes. After switching to water, no desorption was observed (results not shown).

### Discussion

The sorption of PCE on both Expandabeads® and carbon was initially rapid, resulting in a decrease of an average of 75 % of the solute. The maximum capacity was reached in over 90 hours for the beads, regardless of their quantity. The sorption of PCE on carbon seemed more complete and rapid when the higher concentration of PCE was used. This is partially a visual artifact as can be seen in the inset of Fig. 3a, which shows the range from 0 to 0.1 mg/L. The measured concentrations at 6 hours were slightly lower with the higher initial concentration of PCE, but the difference was within experimental error.

The calculated  $K_d$  for Expandabeads® was 12.6 L/g (Fig. 8). A  $K_d$  could not be calculated for the activated carbon because the final concentration of PCE was below the detection limit. It can be estimated that the  $K_d$  would be greater than 256 L/g by using a concentration equal to the detection limit in the calculation. By comparison of the  $K_d$  values, it can be

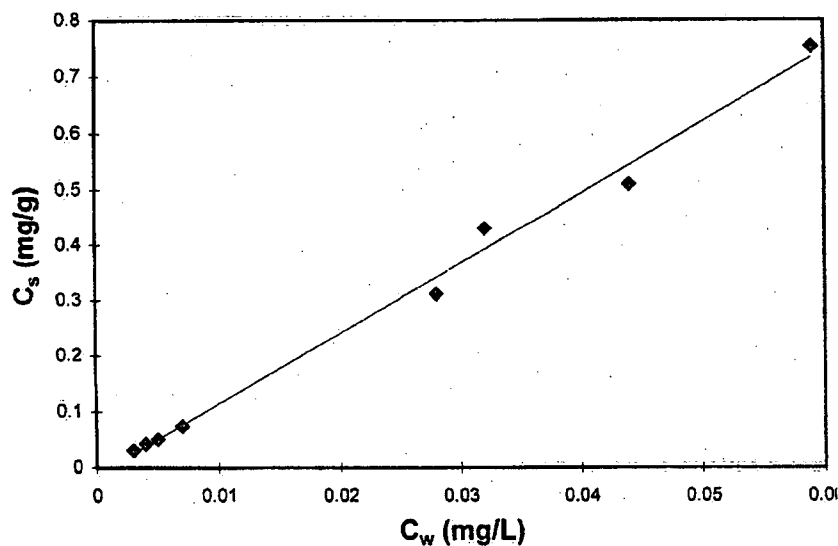


Fig. 8 Sorption isotherm for the batch tests with 1% Expandabeads®.  $C_s$  = mg PCE sorbed per g of beads;  $C_w$  = aqueous concentration of PCE at equilibrium.

seen that the ability of activated carbon to sorb dissolved PCE is far greater than Expandabeads® under these conditions.

The wick tests showed interesting results. During the first 60 hours, the larger amount of wick present resulted in the highest sorption PCE sorbed. After 60 hours, the trend was reversed although the differences between formulations were minimal. This indicates that the wick material (flakes — exact composition not revealed by manufacturer) is a better adsorbent than the polymer and its presence helps to increase the speed of response of the material.

When comparing Imbiberbeads and Expandabeads®, the observations indicate that Imbiberbeads sorb PCE from aqueous solutions faster than Expandabeads®, but both sorb the same amount over time. The products have essentially the same chemical structure but were obtained from two different manufacturers. The only known difference between the two was that the Imbiber Beads® were freshly manufactured whereas the Expandabeads® were stored for several years.

The column tests also indicated that polymer beads do not sorb PCE dissolved in water efficiently. In both columns, breakthrough of PCE occurred rapidly. A retardation factor for Expandabeads® was calculated to be 1.4 for the column containing 1% Expandabeads® and 6.9 for the column containing 10% Expandabeads®. The spikes of PCE concentration observed in the 1% Expandabeads® column was possibly due to pure phase PCE getting into the column. Alternately, they may represent the concentration of solution that has not contacted to the sparsely distributed beads. This type of problem would be very important in a potential field application where the beads would have to be well packed and in high enough concentration to ensure complete contact with the contaminant.

The relatively rapid breakthrough in the two bead-containing columns (Fig. 6 and 7) and the easy leaching observed when the eluent was changed to water indicate that a relatively weak sorption was occurring. It is therefore likely that true absorption into the polymer matrix was not occurring in this case but that the PCE was simply adsorbed on the surface of the beads. This hypothesis is also supported by the batch tests with wick where dilution of the beads with wick did not result in less sorption.

No breakthrough was observed for the column containing 10% activated carbon, after more than 60 pore volumes of 100 mg/L PCE had passed through it. No PCE could be detected in the column effluent even after the addition of four successive additions of pure PCE. While the capacity of charcoal to sorb organic compounds present in water is well known, it was not expected to perform so well with the non-aqueous phase solvent.

If the polymer beads were to be used as an in-situ protective barrier, the rather poor sorption behaviour of the polymeric beads towards dissolved species would require a very thick wall, which would not be cost competitive. In spill situations, the manufacturers recommended to pre-wet the beads with some of the solvents to be cleaned up, thus increasing the "organic nature" of the beads and increasing the rate of the swelling.

It is not possible to envisage such a scenario for an in-situ treatment wall, because environmental authorities are not likely to grant the permission of introducing solvent-containing beads. It would be possible to mix the beads with charcoal, but the added potential protection towards DNAPL movement is not likely to warrant the cost. However, these products may play an interesting preventive role around solvent or light petroleum storage tanks where they could instantly sorb leaking solvents.

### Conclusions

Although Expandabeads® and Imbibers Beads® are marketed as sorbents for pure phase solvents, the batch test results indicated that, for PCE dissolved in water, activated carbon was a better sorbent. This is supported by the large difference in  $K_d$ 's between Expandabeads® and activated carbon. The use of wicking material with Expandabeads® improved the initial sorption rate of PCE but did not increase the total amount sorbed.

The results of the column experiments were consistent with the batch tests. The polymer beads were slow to absorb the PCE and allowed much of the contaminant to escape through the column. Activated carbon, however, sorbed all of the PCE which was pumped through the column and showed no desorption.

Therefore, while the polymer beads visibly soak up pure solvents and are useful for spill cleanups, they would not be suitable for use in permeable treatment beds because of their poor ability to sorb dissolved contaminants. Activated carbon was found to be superior, even in the presence of DNAPL.

### Acknowledgments

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