A laboratory simulation of toluene cleanup by air sparging of water-saturated sands

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Abstract

Laboratory air sparging experiments were performed in narrow acrylic tanks to evaluate the cleanup of toluene in water-saturated sands. Air flow channels in the sediment were identified by way of a colorimetric visualization technique, which allowed pore water samples to be collected at a known horizontal distance from an air channel. Pore water was sampled at periodic intervals during sparging experiments and analyzed by gas chromatography to yield toluene concentration vs. time data. Results indicate that channelized air flow is effective in reducing toluene concentrations in the range of 36–3 ppm, within 2 to 5 days, at least up to 185 mm from an active air channel. While relatively rapid, these toluene reduction times are longer than previously published data, from similar type experiments. The discrepancy is likely a function of air delivery flow rate and proximity of sampling sites to active air channels. Data from the current investigation were used to attempt an estimate of effective diffusion coefficients (D^*) for toluene in clean, well-characterized sands in which the concentration gradient was imposed by sparge air. Calculated D^* values range from 2.98×10^{-8} m^2/s to 5.74×10^{-9} m^2/s, and are significantly faster than previously published values of toluene diffusion in clay soils. However, the values are also slightly greater than diffusion coefficients for toluene in aqueous solutions, indicating that the calculations more likely estimate coefficients of hydrodynamic dispersion (D_L). © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Air sparging is becoming a popular approach towards remediating ground water contaminated with volatile organic compounds (VOCs) and chlorinated solvents. The

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basic concept is to inject air into the saturated zone of a contaminated aquifer. Injected air rises towards the vadose zone (unsaturated zone), scavenging VOCs from the ground water by volatilization. Field studies indicate that ground water contaminant concentrations can be reduced by air sparging [1–5]. Agency directives to remediate sites have caused an increasing number of air sparging field applications, and it is from these experiences that we have gained most of our empirical understanding. Field studies are crucial, but are complicated by several uncertainties, particularly those regarding subsurface geology. This makes the extraction of information on fundamental principles and specific mechanisms difficult.

Only a handful of laboratory investigations of air sparging have been performed [6–10]. Each of these investigations had a different focus. Some focused on engineering system process control [7] and system design [8], while others investigated the relationship of particle size to air-flow geometry [6,10], and the effect of sediment size on area of influence [10]. Laboratory experimental results are essential to a thorough understanding of air sparging, or any remediation technology. The ability to control variables in the laboratory yields data which otherwise could not be obtained. Experimental data are necessary if computer modeling is to play its key predictive role in air sparging applications [11].

While air sparging can facilitate the natural microbial degradation of VOCs by delivering oxygen to the subsurface [12–14], the primary cleanup process of air sparging is volatilization, particularly at sites where dissolved concentrations are > 1 ppm [15]. Volatilization occurs where air flows in the subsurface. Air flow will occur in discrete meandering channels at most sites, with areas of no air flow occurring between individual channels [6,10]. This means that the primary rate-limiting step in the cleanup process is the movement of contaminant to the air channel; and if advective flow is negligible, remediation becomes a diffusion-limited process.

2. Background

Previous experimental studies have investigated the movement of VOCs through sediments. These studies were focused either on the diffusion of contaminants through fine clays [16], with implications for clay-lined waste disposal sites, or on the effects of media characteristics, such as pore moisture content [17].

Only two laboratory studies have examined the movement of VOCs from the standpoint of remediation, and both involved toluene. Because toluene is a common dissolved VOC in petroleum-contaminated aquifers, it is a good representative for laboratory work. Voudrias and Yeh [18] investigated the dissolution of a pool of toluene in water-saturated sands, under laboratory conditions made to simulate a pump-and-treat scenario. Toluene dissolution rates were measured and interpreted in terms of removal efficiency under pulsed vs. continuous pumping. Semer and Reddy [9] performed column and batch tests to assess mechanisms that control toluene removal from saturated soils during air sparging. The latter study specifically evaluated the effect of soil type and injected air flow rate on toluene removal times. The Semer and Reddy [9]

work is the most pertinent to the current investigation, and therefore requires a more detailed description.

Semer and Reddy [9] contained their sediments in cylindrical plexiglass columns 93 cm high and 8.7 cm inside diameter. Separate experiments were performed on fine gravels and Ottawa sand which were contaminated with an initial toluene concentration of either 50 or 250 ppm. Air was injected from the bottom of the cylinder at 6.9 kPa pressure and 380–2225 ml/min flow rate to simulate air sparging. Water samples were collected from ports in the column at periodic intervals during the tests. These samples were analyzed by gas chromatography to generate curves of toluene concentration vs. elapsed time of sparging. Experimental results showed complete removal of toluene in both fine gravels and Ottawa sand. However, complete cleanup took approximately eight times longer in the sand (660 min) than in the gravel (80 min). The significant differences were attributed to the different air flow geometries in gravel vs. sand. It was postulated that pervasive, bubbly air flow observed in the gravels allowed more air–toluene contact, and subsequent volatilization, than did the discrete air channels formed in the sand. Diffusion of toluene to the air channels appeared to be primarily responsible for the increased contaminant removal time in the sand.

In light of the above, the present authors thought that laboratory experiments were needed to investigate the drop in toluene concentration vs. time, at some *known* distance from an air channel, formed within well-characterized, water-saturated sediments. This study reports the findings of such experiments, and uses the data to make a preliminary estimate of effective diffusion coefficients (D^*) for toluene in a water-saturated system in which the concentration gradient is imposed by the flow of air (i.e., an air sparging analogue).

3. Methods

3.1. Apparatus

Experiments were performed in visualization tanks (90 cm high × 90 cm wide × 2.5 cm deep), constructed of 1.6-cm thick acrylic (Fig. 1). Air sparging was simulated by introduction of air through a 2-cm diameter, 3-cm tall, schedule 80 PVC diffuser at the bottom of the tank. The diffuser had six 1.5 mm diameter holes arranged symmetrically around the top edge. The diffuser served as both the air-injection point and the water entry port. Air delivery was controlled with a low-pressure regulator and flow meter. (Fig. 1) Entry pressures for air ranged from 8.3 to 8.9 kPa and represent those pressures required for the air to just overcome the hydrostatic head and capillary pressure, and begin flowing from the diffuser. Experiments were controlled on the basis of entry pressure, with flow being the dependent variable. Flow rates ranged from 1.0 to 1.3 l/min. Actual delivery pressures at a field site will be a function of depth of sparging plus system design and operation. As illustrated schematically in Fig. 1, the front wall of the tank contained 30 holes arranged to form a grid. The holes were fitted with 18-gauge removable stainless steel syringe needles, which served as water sampling ports. The
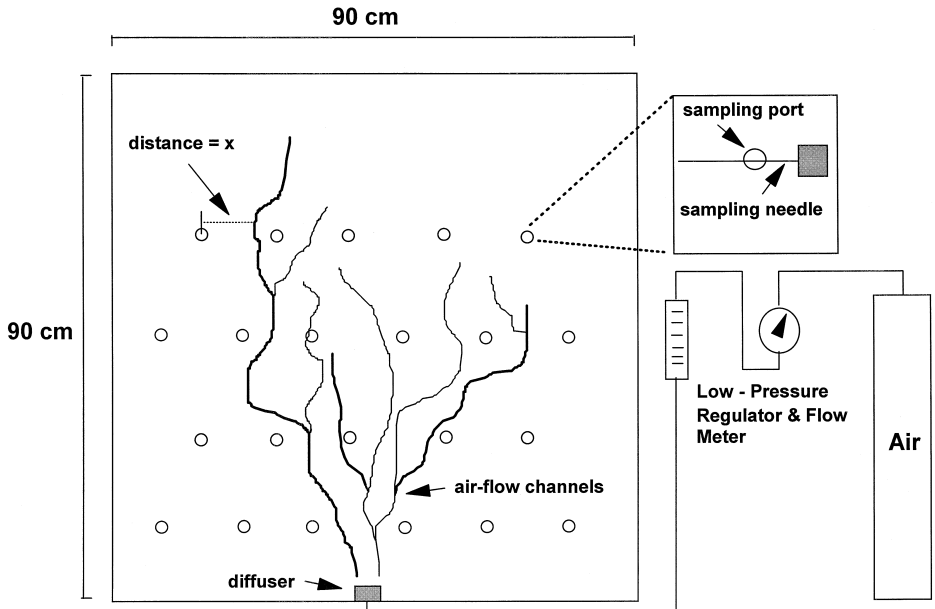
Fig. 1. Schematic showing experimental tank design. Tank was filled with water-saturated sediments, as described in text. Pore water was sampled periodically from a port some distance (x) from an air channel. Grid of sampling ports fitted with stainless steel needles is shown. Diagram is not to scale and the third dimension of the tank is approximately 2.5 cm.

needles were caulked into place and the Luer hubs of the needles plugged with removable Teflon fittings.

A precedent has been set [6–8,10] for performing air sparging experiments in narrow, envelope-like acrylic tanks which yield two-dimensional data. This experimental design is particularly acceptable for measurement of certain intensive parameters, such as concentration [7], which are not dependent on the geometry of the container.

3.2. Sands

Clean, washed natural quartz filter sands were used as the porous medium (Bos Sand, Frankfort, IL, USA). The sands were analyzed by the manufacturer to be 99.40 wt.% SiO_2, plus trace amounts of other inorganic oxides, and virtually zero organic carbon content. Sands were separated on a mechanical sieving machine using ASTM (American Society of Testing Materials) standard mesh sieves. Sand characterization is given in Table 1. Shape analysis (sphericity and angularity) was performed by examining between 100 and 200 individual grains per sand size under the binocular microscope, and classifying them according to the system of Raymond [19]. Porosity values of the sands were determined by multiple measurements of volume displacement in water. Values listed in Table 1 are within the common range for unconsolidated sediments [20], and represent the porosity present during the air sparging test conditions. Hydraulic

Table 1
Sand properties

Grain size (mm) and ASTM pan number	Sphericity	Angularity	Hydraulic conductivity (m/s)	Porosity (%)
> 0.841	51% low	6% v. angular	1.4×10^{-2}[a]	44 ($\pm$1%)
< 1.00	49% high	26% angular	3.3×10^{-3}[b]	
no. 20		25% sub-angular	7.4×10^{-3}[c]	
		21% sub-rounded	2.8×10^{-3}[d]	
		15% rounded		
		7% well-rounded		
> 1.00	46% low	6% v. angular	2.0×10^{-2}[a]	45 ($\pm$1%)
< 1.19	54% high	27% angular	5.0×10^{-3}[b]	
no. 18		27% sub-angular	1.2×10^{-2}[c]	
		23% sub-rounded	3.4×10^{-3}[d]	
		13% rounded		
		4% well-rounded		

[a] Hazen formula [21].
[b] Kruger formula [21].
[c] Zamarin formula [21].
[d] Measured with falling-head permeameter.

conductivities were measured in the laboratory with a falling-head permeameter, and were calculated by way of several published empirical equations relating porosity, grain size and hydraulic conductivity [21]. The measured and calculated values are in good agreement. Calculated values should be considered empirical estimates only; however, they do fall within the upper range expected for clean, unconsolidated sand [22].

3.3. Procedure

To conduct an experiment, sands were loaded into the tank, simultaneously with deoxygenated, deionized water, to form saturated columns. Water was spiked to a known concentration with HPLC-grade toluene before loading. Sands poured into the tank from the top settled through a 1–3 cm column of water during the loading procedure, to ensure saturation. Once loading had commenced, no air was allowed to enter the tank through the diffuser until sparging had begun. A 50-μl sample of pore water was extracted with a gas-tight syringe from one or more sampling ports at the beginning of each experiment, and at regular time intervals during the experiment. (At 45% porosity, this sample is approximately 0.1 cm^3 of the sand column, or about 3 mm radially around the needle tip.) Locations were chosen based on their lateral position relative to an active air channel. Samples were analyzed for toluene concentration with a purge-and-trap gas chromatograph, following U.S. EPA standard method 8020 [23] for analysis of VOCs in water.

During the course of several experiments, blue dye was injected via a sampling port to determine the horizontal rate of flow of water within the saturated sand column. The flow rate in the horizontal direction, directly towards the nearest air channel, was sufficiently slow to be unmeasurable within the duration of the experiments.

3.4. Air flow indicator

During the experiments, air flowed in discrete meandering channels as it rose from the diffuser injection point, up towards the top of the tank (Fig. 1). The collection of these individual channels formed a mosaic pattern in the sand column. This geometry of flow is consistent with previous laboratory observations [6,8,10] of air flow in similar-size particles. Air channels were identified by a colorimetric visualization technique, developed by Peterson et al. [10], which utilizes the color change of iron filings from black to orangish-brown (oxy-iron hydrate or ''limonite'') upon oxidation. Before an experiment, iron filings are in contact with sand and deoxygenated water only. During the experiment, oxygen is provided to the iron filings by the sparge air, but only in the locations where the air pathways are formed in the porous medium, as air migrates from the injection diffuser towards the top of the tank. The orangish-brown color of limonite distinguishes the air flow locations from the other portions of the sediment column, in which the iron filings remain black. This proved to be an effective method to observe the locations and geometry of air flow channels. The elapsed time of sparging required for a very distinct oxidation signal to appear averaged about 12 h, with a less distinct channel appearing within 2–4 h. A fairly crisp boundary between unoxidized iron fragments and oxidized iron fragments (i.e., between air channels and surroundings) could be observed over the scale of less than about 1 cm. Once established, the air channel locations did not change over the duration of the experiments. This method required that the sand columns be spiked in a 1 (iron):7 (sand) volume ratio with reagent-grade iron filings of the same grain size distribution as the sands. Once air flow channels had been identified in an experiment, the horizontal distances (x in Fig. 1) to the nearest sampling ports were documented. Ports selected for periodic sampling were laterally proximal to only one air channel, with the tank wall located in the opposite lateral direction. These horizontal distance values were used in the diffusion calculations described below.

4. Results

4.1. Experimental data

It can be seen from the experimental data listed in Table 2 that a significant decrease in toluene concentration occurs within the timeframe of the laboratory experiments (approximately 2–5 days). In the smaller sand grains (pan no. 20), at a distance 90 mm from an air channel, the toluene concentration was reduced by approximately 8% (2.9 ppm reduction) after the first day, approximately 40% (14.4 ppm) after day 2, and 72% (25.7 ppm) by day 5. In comparison, at a 115 mm distance, the toluene concentration was reduced by about 9% (2.9 ppm reduction) after day 1, about 26% (8 ppm) after day 2, and about 71% (21.7 ppm) by day 5. One day of sparging reduced the concentration 185 mm from an air channel by 26% (5.6 ppm reduction), 2 days reduced it by 71%

Table 2
Experimental data

Grain size (mm) and ASTM pan number	Distance from air channel (mm)	Elapsed time (h)	Toluene concentration (ppm)
> 0.841	90	0	35.7
< 1.00		2.6	31.3
no. 20		22.3	32.8
		28.3	29.6
		47.5	21.3
		118.0	10.0
	115	0	30.5
		4.3	32.4
		23.8	27.6
		45.3	22.5
		51.8	16.4
		119.8	8.8
	185	0	21.9
		4.0	22.1
		23.5	16.3
		29.5	9.6
		49.5	6.4
		119.5	3.0
> 1.00	65	0	19.8
< 1.19		3.3	18.7
no. 18		20.0	3.1
		25.3	3.0
		44.1	2.5
		off 2.2[a]	5.2
	115	0	23.9
		20	21.2
		23.3	14.8
		42.3	13.1
		45.5	15.5
		71.3	15.3
		73.8	13.4
		92.5	12.9
		off 3.5	14.0

[a] Elapsed time after stop of air flow.

(15.5 ppm), and 5 days of sparging reduced it by approximately 86% (18.9 ppm). Similar, but faster cleanup, is observed in the larger-size (pan no. 18) sands investigated. At 65 mm from an air channel, toluene was reduced by 85% (16.8 ppm) in 1 day. After 2 days, the toluene was only reduced by an additional 3% (0.5 ppm). It is important to note that pore water sampled from the same location 2.2 h after the stop of air flow was 2.7 ppm higher in toluene, indicating a movement of toluene back into the region from less-remediated portions of the tank. Toluene concentrations were reduced by 38% (9.1 ppm) within approximately 1 day at 115 mm from an air channel. The concentration increased slightly (up by 0.7 ppm) in the second day, but was reduced by 46% (11 ppm) of the initial concentration by day 4. As observed in the smaller grain experiments, toluene concentration began to rise slightly after cessation of air flow.

Fig. 2 is a subset of Table 2 data and illustrates the reduction in toluene concentration with time at three horizontal distances from an air channel. Curve *A* represents pore water 90 mm away from an air channel formed in pan no. 20 sands. Curve *B* represents water 185 mm away from a channel in pan no. 20 sands. Curve *C* illustrates pore water concentrations 65 mm away from an air channel in pan no. 18 sands. All curves demonstrate an overall reduction of toluene with time, but each has a distinct form. Curve *A* shows an immediate rapid decrease followed by a slight increase in concentration, before a regular decrease occurs. Curve *B* shows a gradual decrease followed by a more typical exponential decrease. Curve *C* shows a significant initial decrease followed by an almost asymptotic reduction in toluene.

4.2. Diffusion estimates

There are several laboratory methods employed to determine effective diffusion coefficients (D^*) in porous media. Shackelford [24] provides an extensive review of the laboratory techniques and the associated analytical equations used to estimate D^*. None of the methods or equations reviewed by Shackelford [24] was developed specifically with air sparging in mind; however, the single-source reservoir method, with decreasing source concentration over time, approximates the experimental simulation of this study. D^* values were back-calculated using Eq. 39 of Shackelford [24]:

$$C/C_0 = \exp\left(nx/H_f + \left(n/H_f \right)^2 D^* t \right) \operatorname{erfc}\left(x/2\sqrt{D^*}\, t + n/H_f \left(\sqrt{D^*}\, t \right) \right),$$

modified for a retardation factor equal to 1, where C = concentration at some time after commencement of sparging, C_0 = initial concentration, n = porosity, t = elapsed time
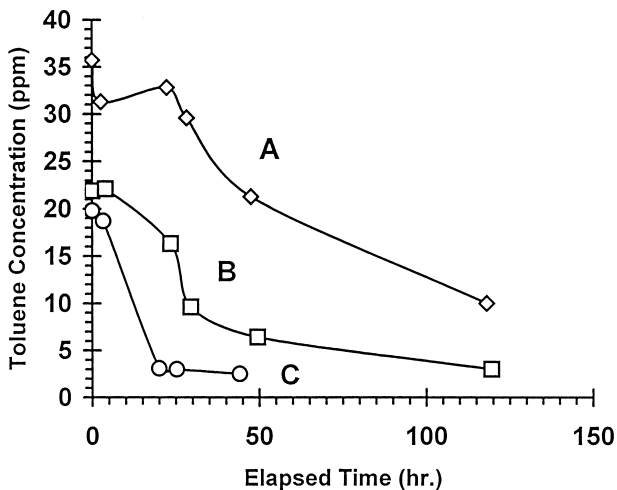

Fig. 2. Pore water concentration vs. time of air sparging experiment. *A* = water sampled 90 mm from an air channel in pan no. 20 (0.841–1.00 mm) sands (Table 1). *B* = water 185 mm from an air channel in pan no. 20 sands. *C* = water 65 mm from an air channel in pan no. 18 (1.00–1.19 mm) sands.

Table 3
Estimated effective diffusion coefficients (D^*) for toluene

D^* (m^2/s)[a]	Concentration range (ppm)	Grain size (mm)	Distance (mm)
1.16×10^{-8}	32.8–10	> 0.841 < 1.00	90
2.98×10^{-8}	22.1–3	> 0.841 < 1.00	185
5.74×10^{-9}	19.8–2.5	> 1.00 < 1.19	65

[a]D^* is from Eq. 39 of Shackelford [24]. If local dispersion is occurring, these values would more accurately approximate coefficients of hydrodynamic dispersion (D_L). See text for discussion.

since the start of sparging, D^* = effective diffusion coefficient, x = distance, H_f = height of water column (source reservoir) above sampling point. The only portions of the curves used in the calculation of D^* were those portions that illustrated a continuous concave-shaped decrease of toluene concentration with time. This means that data collected before 23.5 h for curve B (Fig. 2), and data collected before 22.3 h for curve A (Fig. 2) were not included in the D^* calculations.

The equation used is based on three assumptions: (1) adsorption of toluene to the sand grains is negligible; (2) the effective diffusion coefficient is constant; and (3) toluene movement by advection is negligible. The first assumption is reasonable because the sands used in the experiments were very clean quartz filter sands with virtually no organic matter or clay present. This assumption is consistent with the results of adsorption batch tests by Semer and Reddy [9] which demonstrated that at concentrations of 50–250 ppm, toluene absorption is insignificant in sand-sized grains. Assumption 2 is clearly not the case, since variable diffusion rates can be calculated from the data of this study; however, as Shackelford [24] pointed out, ''When methods which assume constant diffusion coefficients are applied to systems in which such an assumption is not valid, a mean value of D^* is obtained.'' Assumption 3 is based on the blue dye experiments (mentioned in Section 3.3), in which no detectable horizontal movement was observed over the duration of the experiments. Therefore, considering the assumptions, the calculated values (Table 3) are not intended to be a rigorous determination of D^* values, but rather an estimate of the average order of magnitude that could be expected in clean sands.

5. Discussion

5.1. Comparison with previous sparging experiments

The results of this investigation are generally consistent with those of Semer and Reddy [9], by demonstrating the effectiveness of air sparging in reducing toluene concentrations in a relatively short period of time. More specifically, the ''hump'' in concentration vs. elapsed time, as seen in curve A of Fig. 2, was also observed in the

previous sand experiments [9]. Fig. 5 from that publication [9] shows measured toluene concentrations dropping initially, followed by an increase in concentration, in turn followed by a decrease in toluene concentration with elapsed time. An explanation is not given by the authors. We propose that this phenomenon, in their experiments and the current ones, represents an initial stabilization phase for the experiment. It is likely that a certain amount of ''plowing'' of toluene away from the air channels occurs when the air is first injected into the base of the water-saturated soil column. The experiment is not really stabilized until these transient areas of accumulated toluene dissipate. For this reason, early time data were not used for diffusion estimates in the current study. Another important consistency between the two investigations is the shape of the toluene vs. time curve *C* (Fig. 2). The pattern of a significant decrease in concentration initially, followed by an almost asymptotic reduction, was observed by Semer and Reddy [9] in the sparging of fine gravel. The analogous data are illustrated in Fig. 4 of that publication [9].

The main inconsistency between the two experimental studies is the rate of toluene removal. Semer and Reddy [9] observed a drop in toluene from 250 ppm to essentially zero, in 80 min for fine gravel and 660 min for sand. This is significantly faster removal than the fastest observed in this study, in which toluene concentration dropped from 19.8 to 2.5 ppm in 44.1 h (2646 min). One explanation may be that the flow rates of injected air in the previous study were nearly twice the flow rates in the current study. Different operating parameters will result in different removal rates, and may account for the large discrepancy. When Semer and Reddy [9] performed tests on sand with lower initial toluene concentration (50 ppm) and lower air flow rates (960 ml/min), significantly longer toluene removal times were observed. Another explanation may be the proximity of sampling ports to an active air channel or channels. Reduction in toluene concentration could be observed to be very rapid if the sampling port were within a few millimeters of an air channel. The 8.7-cm inside diameter cylinder used by Semer and Reddy [9] almost guaranteed that samples would be collected in close proximity to an air channel. The actual distance was unknown in their experiments, but could not exceed 87 mm, based on the system design. Experiments performed in fine gravel, in which bubbly flow occurs, would be expected to have a faster cleanup time than observed in the current study, in which only channelized flow occurred.

5.2. Comparison with other diffusion data

Estimates of D^* for toluene, calculated from the experimental data of this investigation, are higher than previously published results for toluene and trichlorethylene. For example, Shackelford [24] reported a D^* of 4×10^{-12} m^2/s for toluene in clay till, and Myrand et al. [16] reported values ranging from 3.9×10^{-13} m^2/s to 2.8×10^{-11} m^2/s for toluene in an unweathered glaciolacustrine clay till. This large difference in values is consistent when considering the clean sands of the current study vs. the fine clays of the previous work. A possibly more pertinent comparison can be made with the data of McCarthy and Johnson [17]. They measured D^* values of 4.29×10^{-10} m^2/s for trichloroethylene in water-saturated, no. 8 Ottawa sands. This value is more consistent with the results of the current study, than the clay till results.

The D^* estimates of the current study become problematic when compared with maximum diffusion coefficients for toluene in aqueous solutions, which range from about 5×10^{-10} m^2/s to 9×10^{-10} m^2/s [25,26]. Considering the values accurate to within 10% [26], the effective diffusion coefficients (D^*) for toluene estimated in the present study are 6–30 times greater than those measured in aqueous solutions with no porous medium present. Assuming that the previous aqueous solution measurements are correct, the estimates of the current study indicate that either the equation used by the authors is not applicable for the air sparging situation, or some local dispersion is occurring in the vicinity of the sampling point, meaning that advective transport of toluene is non-negligible. Even though the blue dye tests (mentioned previously) do not indicate horizontal movement of water towards the air channels over the duration of the experiment, some advection may be occurring. Even if limited advection occurs, the calculation made would be more correctly estimating the coefficient of hydrodynamic dispersion (D_L), where D_L equals the combination of molecular diffusion and mechanical mixing [20]. If local dispersion is occurring, the equation used is automatically compromised because its use assumes negligible advective flow.

Further investigation, possibly including a reinvestigation of toluene diffusion in pure aqueous solutions, is necessary to confirm the estimates.

6. Conclusions

The following conclusions can be made from the results of this experimental study: (1) channelized air flow is effective in reducing toluene concentrations (ranging from approximately 36 to 3 ppm) in water-saturated sands, within a 2–5 day time period, at least up to 185 mm away from an active air channel; and (2) effective diffusion coefficients of toluene in clean sands *may* be as fast as 2.98×10^{-8} m^2/s to 5.74×10^{-9} m^2/s when the concentration gradient is caused by channelized sparge air; however, these values are more likely to represent coefficients of hydrodynamic dispersion — a combination of molecular diffusion and mechanical mixing. Until a thorough corroboration is achieved between experimental results, computer modeling and field data on air sparging, efforts to design laboratory tests that elucidate fundamentals of ground water remediation should be continued.

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