



Mobility and sorption of bis-2-chloroethyl ether in an aquifer material

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ABSTRACT

Active treatment of BCEE (bis-2-chloroethyl ether) is being currently performed in the on-site Cohansey Aquifer at the Lipari Superfund Site. Remediation of BCEE in the underlying Kirkwood aquifer is being considered, necessitating investigations of BCEE geochemistry in aquifer material from the site. It is currently unknown to what extent BCEE is present in the dissolved, sorbed, or free-product phase in the Kirkwood Sand aquifer material. A series of partition coefficient sorption, column leach, and column loading tests were conducted to determine BCEE sorption to, and mobility in, the Kirkwood Sand aquifer material. The leach studies indicated that up to 50% of BCEE spiked (as free-phase product) onto two aquifer material column designs could be leached in approximately 18 h, due to the high aqueous solubility of BCEE. Dissolved BCEE concentrations then began to plateau as sorption reactions hindered further leaching, resulting in up to 80% removal after 48 h. Column loading and batch sorption experiments suggest that BCEE mobility is limited by sorption rather than solubility factors. Tracer tests in both column loading and batch sorption tests indicate sorption hinders leaching of BCEE from the Kirkwood Sand material.

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1. Introduction

Aquifer contamination by chlorinated compounds is a common occurrence [1–4]. Field investigations are used to delineate plumes and implement containment or remediation strategies [2,5], whereas laboratory studies are often important for understanding potential transport and remediation mechanisms [4,6,7]. Contaminant sorption to aquifer material may slow remediation efforts [8,9], and therefore can limit its ultimate effectiveness, necessitating a thorough understanding of aquifer-contaminant geochemistry.

The US Environmental Protection Agency (EPA) is actively remediating BCEE (bis-2-chloroethyl ether) in the on-site Cohansey Aquifer at the Lipari Landfill Superfund Site, NJ, and is considering performing BCEE remediation in the underlying Kirkwood aquifer. The Lipari site was contaminated through surface release of nearly 3 million gallons of BCEE, resulting in a contamination area of approximately 6 acres in the underlying aquifer [10]. It is currently unknown to what extent BCEE is present in the dis-

solved, sorbed, or free-product phase in the Kirkwood Sand aquifer material. Bis-2-chloroethyl ether is somewhat soluble semivolatile organic compound (solubility 10.4 g/L, vapor pressure 0.71 mm Hg at 20 °C, Henry's law constant = 1.8×10^{-5} atm m³ mol⁻¹). Existing literature suggests that BCEE should be easily removed from sandy soil, primarily due to its solubility [1,11]; however, remediation in the Cohansey aquifer at Lipari has been slow, suggesting significant retardation may be occurring.

Column and batch tests are often used to provide information about contaminant mobility in solid media [12–14]. The present study was designed to investigate the mobility of BCEE in Kirkwood Sand material by conducting a series of partition coefficient sorption and column leach tests. The work described here is divided into 4 primary efforts: (1) determination of BCEE equilibrium sorption partition coefficient on Kirkwood Sand material, (2) leaching of free-phase-spiked BCEE from intact Kirkwood Sand material, (3) leaching of free-phase-spiked BCEE from slurry packed Kirkwood Sand material, and (4) sorption of dissolved BCEE onto slurry packed Kirkwood Sand material. Research effort 1 is a batch sorption experiment, whereas efforts 2, 3, and 4 involve column studies. The two column leach designs were used to elucidate any effects of channellized flow. The data generated from these 4 experiments suggest that BCEE sorption to the Kirkwood Sand aquifer material could be a factor affecting BCEE mobility more than does aqueous solubility.

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2. Materials and methods

2.1. Reagents and sediment material

All chemicals used were of reagent grade or higher purity and used without further purification; the deionized water used had a resistivity of 18.3 M Ω cm. Neat BCEE liquid (99%) used for sediment spiking was purchased from Sigma–Aldrich (St. Louis, MO), and was used as received. Analytical BCEE standards for instrument calibration were purchased from Restek (Bellefonte, PA). Single element dissolved (1000 mg L⁻¹) lithium standards for tracer spiking and quantitation were purchased from SPEX CertiPrep (Metuchen, NJ). Kirkwood Sand material was collected as a 5.1 cm diameter boring in the field and stored in a 5.1 × 90 cm clear polyvinylchloride (PVC) tube. The Kirkwood Sand material (76.7% sand, 19.0% silt, 4.3% clay) has a pH of 4.9, and contains 3700 mg kg⁻¹ organic carbon and 205 mg kg⁻¹ total nitrogen (dry soil weight).

2.2. Analytical

All BCEE concentrations were determined using an Agilent (Santa Clara, CA) 6890 Gas Chromatograph equipped with a 5973 Mass Spectrometer (GC–MS) following EPA method 8270 [15]. Deuterated 1,4-dichlorobenzene was used as an analytical internal standard for all analyses. The background BCEE concentration in the Kirkwood Sand (approximately 2 mg kg⁻¹) was determined by GC–MS after Accelerated Solvent Extraction (EPA method 3545) [15] using a Dionex ASE 200 (Sunnyvale, CA). BCEE was extracted from liquid samples by adding 1 mL of aqueous sample to 10 mL of hexane in a borosilicate glass vial. The mixture was then shaken for 2 h to allow extraction of the BCEE into the hexane layer. After extraction, 7 mL of the hexane layer was removed and dried using sodium sulfate. The dry hexane solution was diluted as appropriate, spiked with internal standard, and analyzed by GC–MS as described above.

Lithium, used as a tracer in certain experiments as described below, concentrations in the batch sorption and column loading experiments were determined using a PerkinElmer (Wellesley, MA) Elan 6000 Inductively Coupled Plasma–Mass Spectrometer (ICP–MS) equipped with a MiraMist nebulizer, following EPA method 6020 [15]. Samples were prepared for lithium analysis by diluting aqueous samples with 1% nitric acid; the lithium ions at *m/z* 6 and 7 were monitored for quantitation. Scandium was added on-line prior to the nebulizer using a mixing-T and used as an internal standard to correct for instrumental drift.

2.3. Partition coefficient study

To determine the equilibrium distribution of BCEE between water and the Kirkwood Sand material, the partition coefficient (K_d) was measured at pH values of 4, 6, and 10. The pH range was intended to help elucidate pH effects on the sorption mechanism in this system. Approximately 250 g of Kirkwood Sand material was shaken with 1 L of deionized water to remove some of the BCEE already present in the sample from the site so that known amounts then could be spiked onto the material to determine the K_d . The K_d experiment was performed, in triplicate, by weighing 2 g of the water-extracted Kirkwood Sand material into three sets of 4 borosilicate glass vials, into which 12 mL of deionized water (pH 6) was added, containing 0, 8.3, 83, and 833 mg L⁻¹ dissolved BCEE. The experiments performed at pH 4 and 10 used dilute hydrochloric acid or sodium hydroxide to adjust the pH of the solution accordingly. Lithium nitrate in solution was also spiked into the vials at lithium concentrations of 0, 0.42, 4.2, and 42 mg L⁻¹ to determine the lithium K_d on Kirkwood Sand material. Data collected for lithium sorption is used as a comparison value in support of the col-

umn loading study described below. The vials were gently shaken for 24 h to reach equilibrium [16].

After equilibration, the liquid samples were centrifuged to remove the solid material. The supernatant was then analyzed for BCEE and lithium as described above. The amount of BCEE and lithium sorbed to the solid material was calculated by difference. The data for BCEE and lithium were plotted, and the K_d values determined following the linear Freundlich model [16,17].

2.4. Intact column experiment

The intact column experiment used aquifer material from the 5.1 × 90 cm sediment core collected from the site. The column leach apparatus was set up by cutting a 5.1 cm long section from the PVC tube containing the intact core material and placing polyethylene frits on top and bottom prior to adding PVC caps onto the tub ends to seal the system. A high pressure pump was then connected to the column to deliver the deionized water eluent at sufficient pressure to elute through the intact material. The 35 mL min⁻¹ flowrate created significant backpressure, necessitating the use of a high pressure pump. Due to potential BCEE volatility, all experiments were conducted in a laboratory fume hood.

The column was spiked with 1.1 g of neat BCEE, using a syringe, just below the sediment surface and allowed to equilibrate for 18 h before leaching began. After equilibration, the eluent flow was started (35 mL min⁻¹), and effluent samples were collected in high density polyethylene (HDPE) vials when the first drops reached the end of the tubing attached to the fraction autocollector. Forty-nine hourly samples in total were collected for the intact column study and analyzed for BCEE as described above.

Control vials containing dissolved BCEE were placed next to the sample autocollector, to monitor any loss of BCEE due to sorption to the HDPE sample containers or the evacuation of air by the hood vent. To minimize loss due to volatilization in the fume hood, a box was placed over the autocollector samples and control samples. The control samples had BCEE concentrations of 1000 and 10,000 mg L⁻¹ BCEE, and after 24 h the BCEE recovery was 54% in the 1000 mg L⁻¹ sample and 90–99% in the 10,000 mg L⁻¹ samples, indicating BCEE loss by volatilization or sorption is negligible in higher concentration samples, but almost half can be lost in lower concentration samples after 24 h. The analyte recovery for the 1000 mg L⁻¹ is within traditional method quality control limits (35–110%) for BCEE analyses [18], indicating, the column experimental results are valid because the effluent samples were kept in the HDPE vials for less than 14 h before transferring to borosilicate glass vials for extraction and analysis.

2.5. Slurry packed column experiment

For the second column leaching experiment, fresh Kirkwood Sand material was removed from the original PVC tube received from the field site, mixed with water in a 50:50 ratio, and the resulting slurry was packed into new columns. Two clear polycarbonate columns 10.2 cm diameter by 5.1 cm long (Columns A and B) were each slurry-packed using 575 g of Kirkwood Sand material. Because the soils were thoroughly mixed and homogenized prior to packing, and the columns were larger in diameter, the backpressure from these columns was not as high as in the intact column experiment described previously. Therefore a diaphragm pump was adequate to deliver the eluent in these experiments.

Each column was spiked with 1.1 grams of neat BCEE, using a syringe, just below the soil surface and allowed to equilibrate for 18 h before leaching began. After equilibration, the eluent flow was started, and effluent sample collection began when the first drops reached the end of the tubing attached to the fraction autocollector. The flowrate was approximately 35 mL h⁻¹ column⁻¹, with hourly

Table 1
Partition coefficients (K_d) and correlation coefficients (R^2) determined for BCEE and lithium on the Kirkwood Sand material.

Replicate	A	B	C	Average	K_d , std. dev.	A	B	C
Analyte	K_d , (mL g ⁻¹)			K_d (mL g ⁻¹)		Corr. Coeff. (R^2)		
BCEE (pH 4)	0.6	0.6	0.7	0.63	0.050	1.000	1.000	0.997
BCEE (pH 6)	0.6	0.5	0.6	0.57	0.058	0.993	0.998	0.998
BCEE (pH 10)	0.7	0.5	0.5	0.56	0.086	1.000	0.818	0.776
Lithium (pH 4)	0.4	0.3	0.1	0.28	0.146	0.995	0.996	0.995
Lithium (pH 6)	0.5	0.4	0.4	0.43	0.058	0.994	0.998	1.000
Lithium (pH 10)	0.4	0.2	0.1	0.23	0.140	0.996	0.902	1.000

samples collected for 46 h. Each sample was analyzed for BCEE as described above.

2.6. Column loading study

The third column experiment used the slurry packed columns described above (Columns C and D). After the 46 h leaching period for which BCEE leach concentration data was collected, the columns were leached with water for an additional 48 h (additional 1600 mL), such that the effluent BCEE concentration was negligible. The eluent pumped through the columns was then changed to deionized water containing approximately 250 mg L⁻¹ BCEE and 50 µg L⁻¹ ⁶Li nitrate (as a tracer) at a flow rate of approximately 15 mL h⁻¹ column⁻¹. This eluent was then pumped through the columns for 50 h. The breakthrough of BCEE, compared to that of the conservative lithium tracer, would determine if BCEE sorbed to a greater extent to the Kirkwood Sand material than did lithium. Effluent BCEE and lithium concentrations were determined by GC-MS and ICP-MS, respectively, as described above.

3. Results and discussion

The water solubility of BCEE over a specified temperature range, specifically 24, 48, and 74 °C (nominally 25, 50, and 75 °C) was investigated previously, with an equilibrium (10 day) solubility of ~13,000 mg L⁻¹ determined at 24 °C, and approximately 8% and 20% higher values at the elevated temperatures, respectively [19,20]. The relatively high solubility of BCEE is a critical factor affecting the potential leachability from the Kirkwood Sand material. If column effluent concentrations are less than the solubility limit, it is likely that sorption processes are at least partially responsible for the observed reduction in concentration.

3.1. Partition coefficient study

The partition coefficient (K_d) quantifies the equilibrium distribution of BCEE and lithium between the Kirkwood Sand material and water [16]. The partition coefficients and linear correlation coefficients of the regression fit for the triplicate K_d determinations at each of the three pH values tested are given in Table 1. Linear correlation coefficients for the regression lines used to calculate the K_d values are very close to one, supporting the use of the linear Freundlich isotherm model for this system. The K_d for lithium is just slightly lower than that of BCEE, suggesting that it sorbs less than BCEE to the Kirkwood Sand material at all pH values tested, and therefore acts as a conservative tracer for these tests. The sorption information provided by the K_d is useful in understanding the column loading study described below, in which lithium is not retained as strongly in the soil, and approaches an equilibrium concentration faster than BCEE, however, partition coefficients of less than 1 mL g⁻¹ represent minimally retained compounds on the solid surface.

As a comparison, metals such as arsenic and uranium can have K_d values of 10–1000 mL g⁻¹, depending on the system [17]. The

average K_d for BCEE determined in our study is 0.57 mL g⁻¹ (pH 6; the average for all three pH values is 0.59 mL g⁻¹), compared to other common halogenated organic compound K_d values ranging from 0.1 to 5 mL g⁻¹, reported in the literature [12,21–23]. Table 2 lists partition coefficients reported for several other halogenated organic compounds on various solid substrates.

The BCEE K_d values determined in this experiment are in agreement with values for other halogenated organic compounds reported in the literature for a wide range of solid materials. The molecular structure of BCEE is such that the terminal chlorines and the ether group contribute some polar characteristics to the molecule which could interact with charged soil particle surfaces. For example, at pH conditions below ~6, many metal oxides will have positive surface charges, and therefore may contribute to BCEE sorption, however, the pH range tested in the current study suggests that hydrophilic interactions with charged surfaces will not significantly affect BCEE sorption in this system.

Additionally, the BCEE molecule has hydrophobic character, and therefore may interact with natural organic matter in the Kirkwood Sand material (3700 mg kg⁻¹ organic carbon) [3,12]. BCEE has an aqueous solubility greater than 10,000 mg L⁻¹, and an octanol–water partition coefficient (K_{ow}) of 19.5 [20]. Using the approximation method of Lyman et al., [17,24], an organic carbon partition coefficient (K_{oc}) of 2.1 mL g⁻¹ can be calculated. A K_{oc} of 2.1 mL g⁻¹ suggests at least some affinity for the soil organic carbon phase, although not to the degree of more hydrophobic compounds, such as polychlorinated biphenyls [17].

3.2. Intact column experiment

The concentration of BCEE in the effluent of the intact soil column was plotted versus elution volume (over 49 h), as shown in

Table 2
Comparison of halogenated compound K_d values reported in the literature.

Compound	K_d (mL g ⁻¹)	Material ^a
BCEE ^b	0.57 ± 0.058	KS
1,2-Dichloroethane	0.09	PS
1,1,2,2-Tetrachloroethane	0.13	PS
1,1,2-Trichloroethane	0.26	PS
1,4-Dichlorobenzene	0.34	PS
2,4-D ^c	0.62	CS
Ethylene dibromide	0.73	CS
Atrazine	3.24	CS
Chlorobenzene	3.96	CS
Methylene chloride	0.23	S-B
Trichloroethylene	0.52	S-B
Pentachlorophenol	4 ± 4	AS
2,4,6-Trichlorophenol	3 ± 3	AS
2,3,4,6-Tetrachlorophenol	5 ± 8	AS

^a KS, Kirkwood Sand (pH 6, 76.7% sand, 19.0% silt, 4.3% clay); PS, Pleistocene Sand (77.5% sand, 9.4% silt, 13.1% clay; [21]); CS, Capac Soil (54.6% sand, 24.0% silt, 21.4% clay; [22]); AS, aquifer sediments [12]; S-B, soil-bentonite (soil 0.6% gravel, 96.8% sand, 2.6% silt and clay [23]).

^b Current study.

^c 2-Chloro-4-ethylamino-6-isopropylamino-s-triazine.

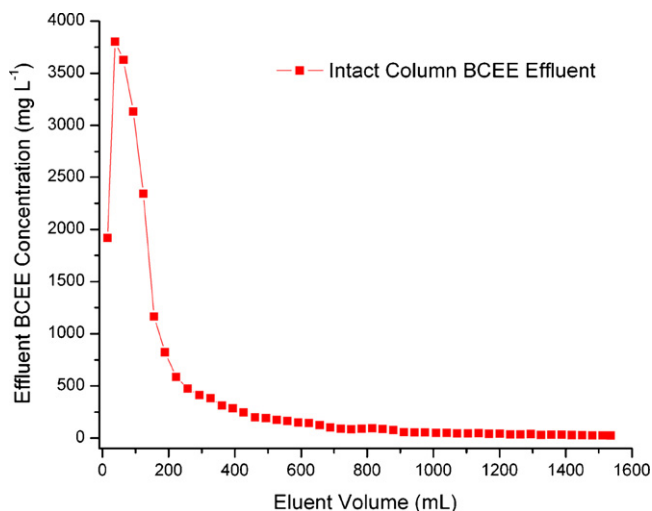


Fig. 1. Concentration of BCEE in intact column effluent samples over 49 h.

Fig. 1. There was an initial spike of BCEE (~3800 mg L⁻¹) that eluted from the column followed by a steady decay in concentration that approached a plateau value near 40 mg L⁻¹ after 800 mL have eluted (approximately 24 h). The maximum concentration measured is only about one third of the aqueous saturation value for BCEE.

A total of 1.1 g of BCEE was spiked onto the column prior to the leaching study. The mass of BCEE leached from and remaining in the column material is plotted in Fig. 2. As the leaching continues, the amount of BCEE remaining in the column slowly decreases, with approximately 50% removed after 18 h (~550 mL). Approximately 60% of the spiked BCEE is removed after 49 h of leaching (almost 1600 mL eluent volume). The leveling off of the leached amount is likely due to natural preferential flow paths of the water through the intact material. Because of the tight packing of the sand grains with silt, there is likely some preferred channelization, possibly along the column walls, that inhibits uniform leaching of the material in this column design.

However, the trends observed in Figs. 1 and 2 could be the result of the initial overloading of the aquifer material's sorption sites. The initial elution of BCEE represents 'excess' compound that could not be retained due to site saturation, or was retained by weak interactions with the solid material. Therefore, the long decaying effluent

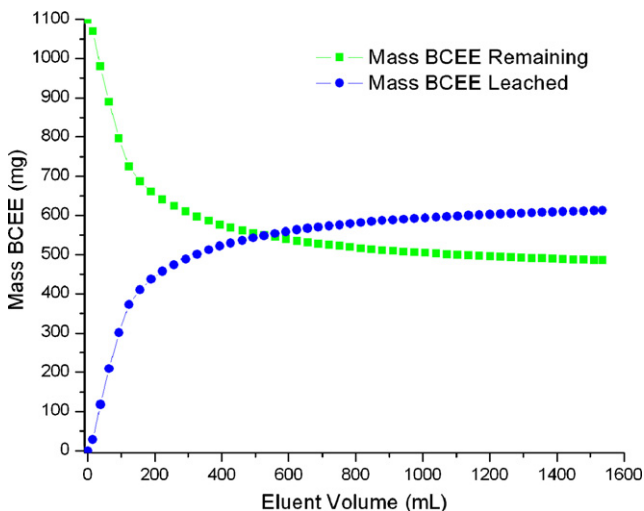


Fig. 2. Mass of BCEE remaining in and leached from the intact soil column.

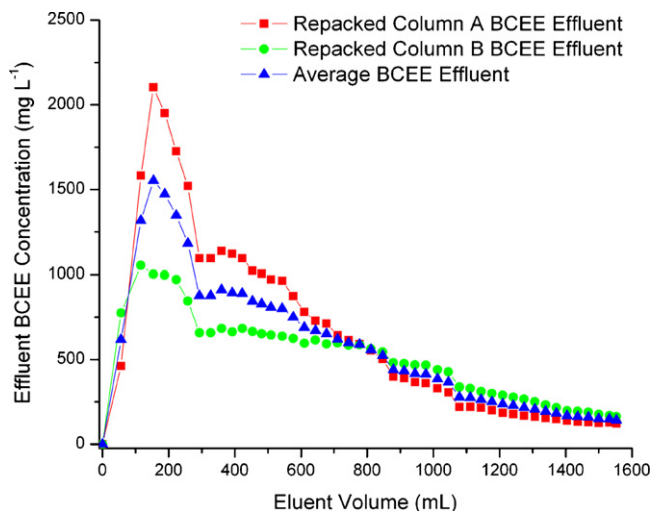


Fig. 3. Concentration of BCEE in each slurry packed column effluent sample.

concentration tail could be indicative of slow release of sorbed BCEE from the aquifer material as the leaching progresses.

3.3. Slurry packed column experiment

Because of the possibilities of channelized flow in the intact experiment described above, column leach experiments using slurry-packed columns were performed to provide a more homogeneous media to reduce the likelihood of non-uniform flow. The BCEE concentration of each effluent from the slurry packed columns (A and B) was plotted versus elution volume (over 46 h) as shown in Fig. 3. Again, there was clearly an initial spike of BCEE (average ~1500 mg L⁻¹) that eluted from the columns followed by a steady decay in concentration. Columns A and B behaved similarly to each other, and to the previous intact column, over the time period measured; the average effluent concentration for columns A and B is also shown in Fig. 3. The effluent concentrations reach a plateau value near 150 mg L⁻¹ after the 46 h leach period (1600 mL). Again, the maximum dissolved BCEE concentration detected was well below the aqueous solubility limit.

The mass of BCEE leached from and remaining in the slurry packed columns is plotted in Fig. 4. As the leaching progresses,

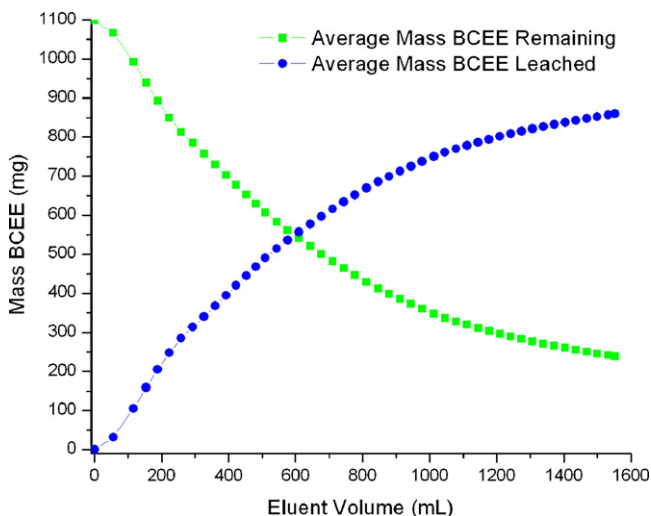


Fig. 4. Average mass of BCEE remaining in and leached from the slurry packed columns.

the amount of BCEE remaining in the column slowly decreases with approximately 50% removed after 16 h, slightly earlier than the intact column experiment shown in Fig. 2. The total amount of BCEE removed from columns A and B after 46 h of leaching was 82% and 75%, respectively, yielding an average of 79% removal. The higher removal efficiency of the slurry packed columns compared to the intact column could be due to the more uniform flow through the homogenized material in the slurry packed columns. The long tail again suggests that sorption processes are hindering complete removal of BCEE from the aquifer material, as the effluent concentration is well below solubility limits.

The intact column study suggested that preferential flow paths may affect BCEE leaching in this type of column design. There was a steady decrease in the BCEE concentration which leveled off after 800–1000 mL were eluted through the column. There was no chemical difference between the intact column study and the slurry packed columns, however, the physical difference caused by sample homogenization and slurry packing did result in more effective leaching of BCEE from the slurry packed columns. If sorption to the intact material, alone, was responsible for the difference in total BCEE removed between the two column designs, one would expect more effective leaching from the intact column due to the lower sediment mass (5.1 cm diameter for intact column, compared to 10.2 cm in the slurry packed columns). However, because less is leached from the intact column, it is likely that heterogeneous flow patterns reduced the leaching efficiency.

3.4. Column loading study

A column loading study was performed to determine if the Kirkwood Sand material has a finite capacity for BCEE sorption, and could continually remove BCEE from solution until the sorption sites are saturated. If equilibrium sorption occurred quickly, measurable effluent concentrations would be observed once all sorption sites were filled. BCEE and lithium were simultaneously eluted through the columns (250 mg L^{-1} and $50 \text{ } \mu\text{g L}^{-1}$, respectively) such that any retardation or concentration suppression of BCEE eluted from the columns would be measured relative to the lithium tracer.

The average fraction of BCEE and lithium analytes in the column effluents compared to the eluent as a function of elution volume is shown in Fig. 5. There is a delay in observed concentrations of both analytes as eluent is pumped through the soil column, because of

the initial column flushing with water. The concentrations of BCEE and lithium slowly increase during the loading period, and after 50 h (over 800 mL), the lithium concentration is within 85% of the eluent initial spike concentration, whereas the BCEE is only about 58% of the eluent concentration, indicating that sorption to the Kirkwood Sand material is retarding BCEE migration through the column, relative to lithium. Fig. 5 illustrates that neither species is completely retained, although BCEE sorbs to a greater extent than does lithium.

The dip in BCEE concentration observed at approximately 425 and 775 mL elution volume is due to the overnight sample collection in the HDPE vials. As described previously, the control samples placed next to the effluent samples showed sorption losses of 1–46% (over 24 h), depending on concentration. Accounting for possible loss of BCEE due to the sample collection vials, the BCEE ratio could be approaching 70%, however, the net result is a suppression of BCEE concentration in the column effluent possibly due to sorption in the Kirkwood Sand material. To facilitate comparison to the leach results presented in Figs. 1–4, the data plotted in Fig. 5 have the same x-axis scale.

4. Conclusions

The data provided by the column leaching studies indicate that BCEE will leach from the Kirkwood Sand, although sorption can hinder complete removal. This result was demonstrated by the column loading study in which BCEE eluent concentration fractions remain below that of the lithium tracer. Two types of leach columns were tested, intact materials received from the field and slurry-packed material. Both column designs showed some retention of BCEE in the aquifer material, although the slurry-packed columns may have provided more uniform flow through the material, with ultimately more of the initially added BCEE material being leached from the slurry-packed columns.

The partition coefficient, K_d , was also determined under three pH conditions for BCEE and lithium. Results of the sorption coefficient study support the column loading data where BCEE is sorbed to a greater extent than is lithium. The K_d calculated from the batch sorption experiment is in the range of other halogenated compounds reported in the literature. Reported K_{ow} and calculated K_{oc} values indicate that hydrophobic sorption reactions are likely important, as pH changes did not significantly affect BCEE sorption. Although charge density estimations suggest that hydrophilic sorption reactions may also be involved, the sorption results indicate that hydrophobic interactions dominate in this system.

The sorption and leaching data presented support field observations that BCEE remediation is slower than predicted from BCEE solubility data. Sorption of BCEE to the Kirkwood Sand aquifer material is partially responsible for BCEE retardation although physical flow characteristics shown in the intact column leaching experiments can also affect BCEE leaching. The partition coefficient study performed with lithium as a comparison supported BCEE sorption to the aquifer material, which further suggests retardation of BCEE relative to more conservative species.

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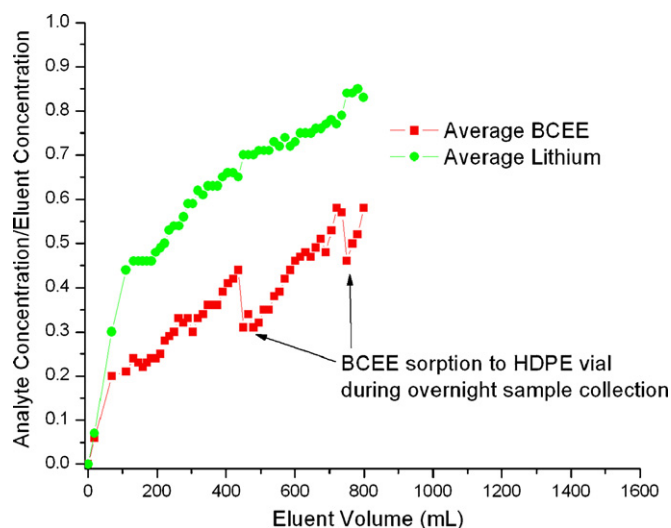


Fig. 5. Average fraction (C/C_0) of BCEE and lithium to initial eluent concentration in column effluent.

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