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AN EVALUATION OF PHYSICAL PROPERTIES OF POLY VINYL ALCOHOL BEADS FOR USE AS PERMEABLE BARRIER FOR GROUNDWATER REMEDIATION

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Nomenclature

TCE	Trichloroethylene
PVA	Poly Vinyl Alcohol Beads
c-DCE	cis-Dichloroethylene
CAH	Chlorinated Aliphatic Hydrocarbons
mL	Milliliters
L	Liters
°C	Degree Celsius
°F	Degree Fahrenheit
g	Grams
mg	Milligrams
lb	Pounds
ft	Feet
µg	Micrograms
rpm	rotations per minute
COD	Chemical Oxygen Demand
psi	Pounds per square inches.
GC	Gas Chromatograph
FID	Flame Ionization Detector
PID	Photo Ionization Detector
ECD	Electron Capture Detector
USEPA	United State Environmental Protection Agency

1 Introduction

1.1 Background

A permeable reactive barrier is a wall built in the subsurface where a contaminated plume has to move across the barrier, as shown in Figure 1-1 (US EPA, 1998). The permeable reactive barrier will not obstruct or be a barrier for water flow (Day et al., 1999). Theoretically barrier will react with contaminants, and the water flowing out of the permeable barrier will be free of contamination or the contaminants will be converted to less toxic chemicals. The contaminant removal can be physical, chemical, and or biological including precipitation, sorption, oxidation/reduction, fixation, or degradation (McGovern et al., 2002). There are two basic types of barrier walls that can be installed in subsurface, the funnel and gate type and the continuous wall type.

The funnel and gate design consists of low hydraulic conductivity walls (funnel) and a permeable wall (gate) in between the funnels (Figure 1-2). The funnel modifies the flow pattern and directs the contaminated groundwater to flow towards the gate, where the contaminants are acted up on by various treatment technologies (McGovern et al., 2002). The funnel or the cut off walls are usually slurry walls, sheets piles, or soil admixtures etc. (McGovern et al., 2002).

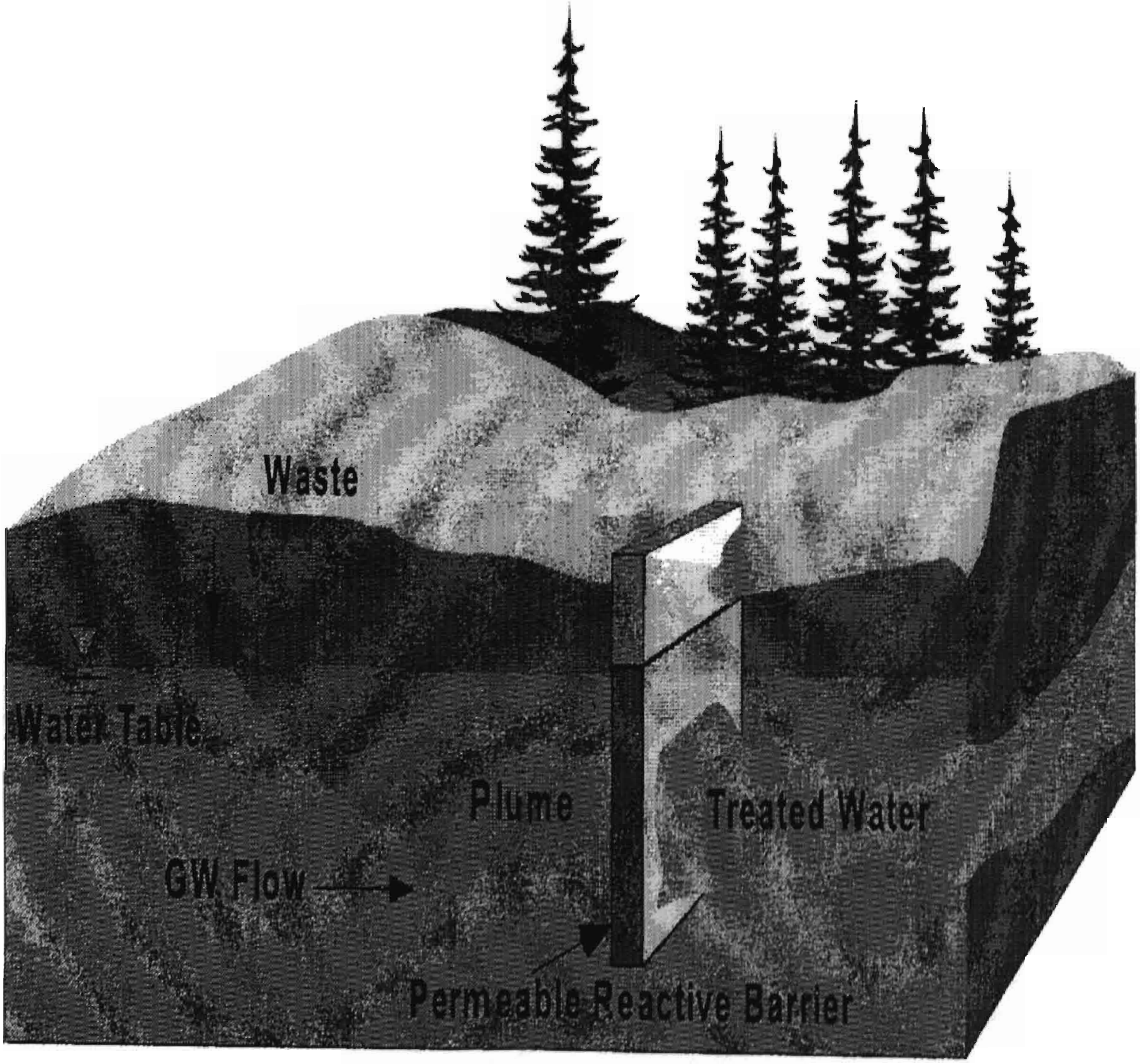

Figure 1-1 Permeable Reactive Barrier (US EPA, 1998)

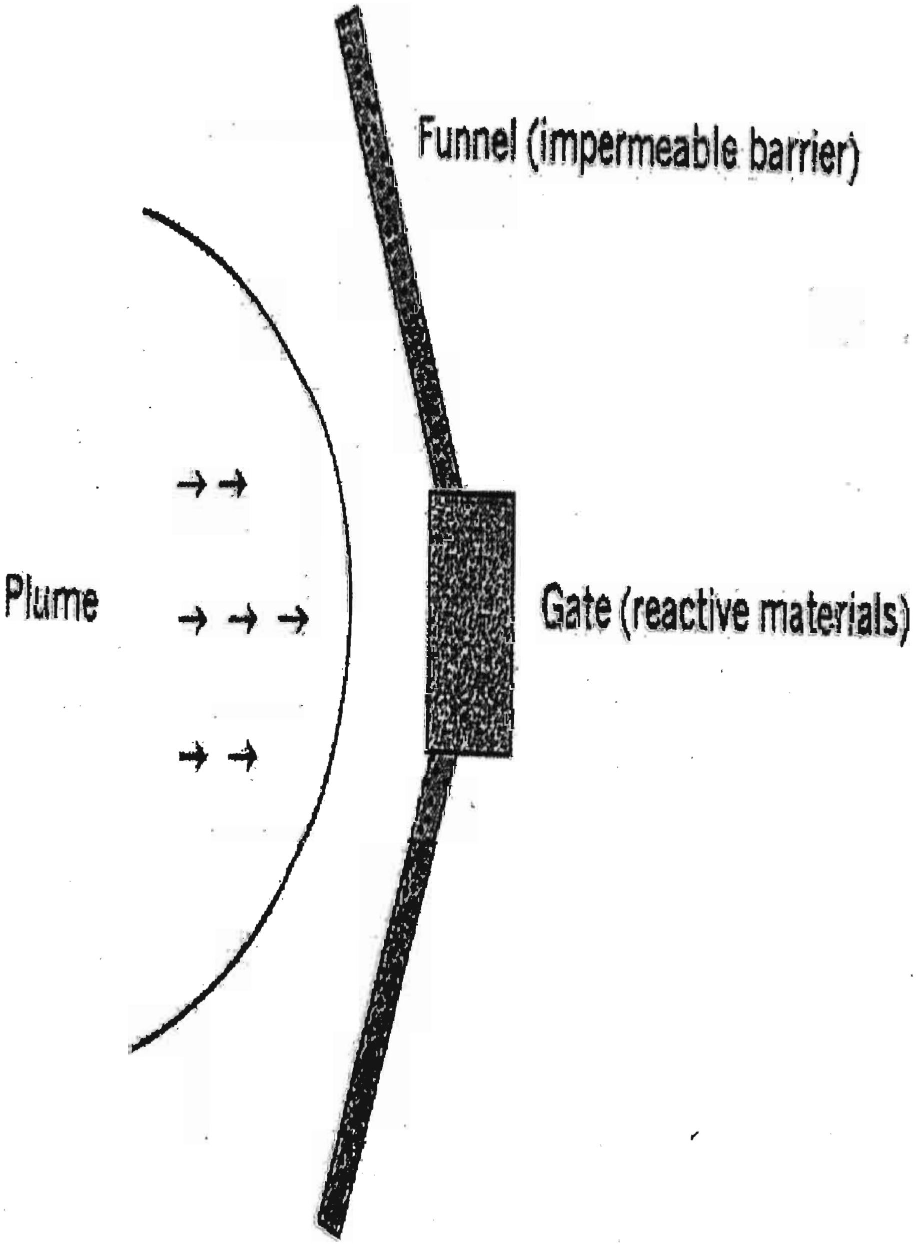

Figure 1-2 Funnel and Gate Design

The continuous wall type barriers are the simplest and generally extend to width and depth of the contaminant plume (Day, 1999). The funnel and gate design could impact the groundwater flow velocity and direction due to its design whereas the continuous wall method will have a lower impact on the groundwater flow (Steven et al., 1999).

1.2 Objectives

The objective of the study was to determine the suitability of poly - vinyl alcohol (PVA) beads to function as a permeable barrier technology for groundwater treatment. The basic emphasis was to study the physical and chemical properties of the PVA beads. The physical properties such as consolidation, permeability and density were studied to check the ability of the PVA beads to withstand the overburden soil pressure and to allow water to flow across it. Adsorption and diffusion tests were also conducted to study the loss of contaminant due to adsorption on the beads and to estimate the quantity of contaminant that diffuses into the beads and also the rate of diffusion. The amount of contaminant diffusing into the beads could be an important parameter, as that is most likely to be the quantity available to the microorganisms, if PVA beads are used as a bio - barrier. The adsorption and diffusion studies were done for trichloroethylene (TCE) and cis-dichloroethene (c-DCE).

2 Literature Review

2.1 Introduction

An effort has been made to study the past research experiences of permeable barrier in groundwater treatment with a particular emphasize given to TCE and c-DCE as contaminants. The applications of permeable reactive barriers in treating various contaminants are presented for the understanding of the efficiency of permeable reactive barriers. Various methods of making PVA beads are discussed. The physical and chemicals properties of a permeable barrier and the efficiency in treating given contaminants are presented for the better understanding of the application of the permeable barrier technology. Various methods of designing the permeable reactive barriers, construction of permeable barriers and their application in groundwater treatment are discussed in detail. The analytical methods for the analysis of TCE and c-DCE are also discussed in detail.

2.2 Applications of Permeable Barriers

Araujo and Teideira (1997) studied the efficiency of alginate - calcium chloride beads for chromium removal by adsorption under varying temperature and pH conditions. The authors found that the temperature range of 10 to 27 °C favored trivalent chromium adsorption onto beads. The optimal pH range based on the authors research was 2 to 4 and adsorption increased with increasing pH.

The authors concluded that adsorption of chromium onto alginate – calcium chloride beads was dominantly due to ion exchange between calcium and chromium at lower concentrations. The authors also suggested that, for the removal of higher concentrations of chromium, an alginate solution can be mixed with a highly concentrated chromium solution and then dropped into calcium chloride to increase the adsorption of chromium onto the beads.

Vogen et al, (1997) studied the performance of a permeable reactive barrier filled with granular iron. The objective of this study was to remediate a trichloroethylene contaminated site. The authors successfully conducted a pilot study as well as a field study on the application of a granular iron reactive barrier as a treatment technology. They did not find microbial fouling in their permeable wall during the two year study period. The permeable barriers were able to reduce the concentration of TCE from 189 mg/L to less than 1 mg/L with an estimated TCE half life of less than 4 hours. The half life was defined by the authors as the time taken to reduce 50 % of the initial concentration. The following (Figure 2-1) degradation pathway was proposed by the authors.

$$3Fe^0 \rightarrow 2Fe^{2+} + 4e^-$$
$$3H_2O \rightarrow 3H^+ + 3OH^-$$
$$2H^+ + 2e^- \rightarrow H^2$$
$$R-Cl + H^+ + 2e^- \rightarrow R-H + Cl^-$$

$$3Fe^0 + 3H_2O + R-Cl \rightarrow 2Fe^{2+} + 3OH^- + H_2 + R-H + Cl^-$$

Figure 2-1 Iron Reduction of Trichloroethylene

2.3 Preparation of Beads

Araujo and Teideira (1997) prepared beads by dropping a 2 % sodium alginate aqueous solution into a 2% calcium chloride solution. The calcium chloride solution was continuously stirred while the alginate solution was delivered into the calcium chloride solution. The alginate beads were cured in the calcium chloride solution for 24 hours and then washed three times with distilled water. The beads were stored in 2% calcium chloride solution at 4°C. The beads were again washed three times before using them in experiments. The authors determined the percentage of alginate in each beads to be 5.6 %, the mean diameter of beads as 2.58 mm, the mean weight of the beads as 9.3 mg, and the solids content as 6.2%.

2.4 Physical Properties of Beads

2.4.1 Gel Strength

According to Vogelsang et al. (1997), gel strength is defined as the constant load required to achieve 0.1 mm compression when applied at the rate capable of compressing the beads by 0.02 mm/s. The authors conducted the gel strength experiment using a gel meter built by Professor A. Elgsater (Norwegian Biopolymer Laboratory, Department of Physics, NTNU, Norway), which was described by Matinsen et al. (1989).

These authors found that the gel maintained its physical integrity in a continuous flow reactor fed with domestic wastewater for more than 2 years, when they did not contain entrapped cells, but had low gel strength. The gels were made of 5% PVA-SbQ, 2 % alginate solution and 0.10 M $CaCl_2$. The fresh gels had strengths of 800 ± 150 mg and the gel beads from the nitrification reactor taken on day 240 had strength of 80 ± 10 mg. The authors compared these results to the work by Hertzberg et al. (1995), where the bead strength was found to be 77 ± 3 mg, which was similar to the strength obtained by the authors. Vogelsang et al. (1997) used the same method as Hertzberg et al., 1995 to determine the bead strength.

PVA – SBQ beads utilized by Hertsberg et al. (1995) were made by a Na- citrate treatment where the alginate was removed by dialysis. From the experimental results of Vogelsang et al. (1997) and Hertzberg et al. (1995,) it can be observed that the gel strength of beads are less in the absence of alginate. Hertzberg et al., 1995 removed alginate by dialysis and recorded a low gel strength of 77 mg.

The low gel strength observed by Vogelsang et al. (1997) in the nitrification reactor was due to alginate removal by the nitrification process.

In the experimental work by Shen et al. (1993) PVA gel cubes were made of 6% PVA by standard freezing and boric acid methods. These beads were fragile and broke down after 2 days of incubation even in closed non – stirred vessels. As a result authors increased the PVA content to 12% to obtain more stable beads.

2.5 Design of Permeable Reactive Barriers

McGovern et al. (2002) used the funnel and gate technology to treat groundwater contaminated with petroleum hydrocarbons. The funnel gate system consisted of an impervious barrier membrane, which directed the contaminant to flow towards the permeable gate zone. The funnel was designed in such a way that it intercepted the contaminant plume downgradient of the spill site and also in adjacent areas where the plume could have migrated. High density poly - ethylene was used as the funnel to direct the contaminated water towards the gate. The gate, the permeable portion, consisted of an air sparging unit followed by a peat layer. The air sparging system was placed at the front end of the gate to bubble air through the system to enhance aerobic biodegradation in the peat layer.

The authors were able to achieve overall removal efficiency of 72 % during a 10 month operational period. According to Gavaskar (1999), the design of a permeable barrier largely depends on the suitability of the site for the application of permeable barrier technology, half lifes of contaminants of interest, local configuration, longevity, monitoring and cost.

Thompson (1996) studied the potential of PVA beads as a bio – barrier for the remediation of chlorophenols. Thompson (1996) successfully immobilized microorganisms in PVA beads and proved that chlorophenols can be biodegraded by immobilized organisms. A column study was conducted by Thompson (1996) to simulate a bio - trench with PVA beads immobilized the microorganisms as a permeable reactive barrier. Batch studies were also conducted to characterize the beads by physical properties such as density, porosity, permeability and compressibility. Chemical properties such as adsorption and diffusion were also evaluated by Thompson (1996). A batch study was also conducted to study the degradation kinetics of chlorophenols by free cells as well as immobilized cells.

The column study by Thompson (1996) was run for 45 days and 100 % trichlorophenol (TCE) removal was achieved within first 14 days. The performance of immobilized beads remained consistent for the 45 days study period. Thompson (1996) also found that the microorganisms immobilized in PVA beads exhibited a lag time of 96 hours and after the lag time, immobilized organisms were able to degrade 10.0 mg/L of TCP in 5.0 hours. The TCP diffusion coefficient into the PVA beads was reported as 3.1 E-6 cm^2/sec. Thompson (1996) also found that the compressibility of PVA beads were similar to that of soft clay, and the porosity and permeability were comparable to coarse sand and gravel. Thompson (1996) also found that the TCP adsorption capacity on PVA beads was low in the order of 5.01 E -15.

2.6 Analytical Methods for TCE and c-DCE

Analytical techniques for the measurement of TCE and c-DCE were studied and are shown in Table 1.0. From reviewed literature, the electron capture detector (ECD) was found to be the most suitable detector for analysis of chlorinated compounds.

Table 1, Analytical Methods for TCE and C-DCE Measurements

Author	Technique	Operating Condition
Duba, A.G., et.al.,1996	Purge and trap, Gas Chromatography (GC), EPA method 601.	Detection limit for Trichloroethene (TCE) 0.20 ppb. Chlorobenzene was used as surrogate standard and the mean recovery was 97%.
Degraffenreid, N. and Shreve, G.S., 1998	Perkin Elmer Gas Chromatograph equipped with Electron Capture Detector (ECD) and HS –40 headspace analyzer was used to TCE analysis.	Chromatographic separation was achieved with DB - 5 capillary column (0.32 mm I.D.), helium was used as the carrier gas at a flow rate of 0.50 mL/min and nitrogen was used as make up gas at 38.50 mL/min.
Kao, C.M. and Lei, S.E., 2000	Degradation product analysis was performed in	Not available.

	accordance with US EPA method 601 using Tekmar Purge- Trap, Model LSC 2000 with a Varian model 3800 GC.	
Inguva, S. and Shreve, G.S., 1999	TCE analysis was done by EPA method 8010A using GC equipped with ECD.	The detection limit for TCE ranged from 0.01 ppm to 2.00 ppm. Chromatographic separation was achieved with DB 5ms capillary column (0.32 mm I.D.) Helium was used as the carrier gas at a flow rate of 0.50 mL/min and Nitrogen was used as make up gas at 38.50 mL/min.
Leahy, J.G., et al., 1996	Pentane extraction method with Flame Ionization Detector (FID) was used for TCE analysis	Rtx-624 silica capillary column (30 m * 0.53 mm * 3 µm) was used for chromatographic separation. Nitrogen was used as the carrier gas. The injection temperature was set at 170°C, the column temperature was set at 65°C and the detector temperature was set at 170°C. The detection limit for TCE was 2.4 µm
Kao,C.M. and L.Yang, 2000	TCE and by - products were analyzed in accordance to EPA method 310, using Tekmar Purge and Trap Model LSC 2000 with Varian model 3800 GC	Not available.
Duhamel, et al., 2002	Chlorinated ethenes, ethane, methane and ethane were analyzed by HP - 5890 GC equipped with FID	Chromatographic separation was achieved in a GSQ PLOT column (30 m* 0.53mm I.D.). The oven temperature was set at 35°C for 2 min to elute methane and ethane, and then increased to 180°C at 60°C/min and finally helds for 4 min at 180°C.

Yu and Semprini., 2002	TCE, cDCE, VC, ethylene and methane were measured by HP-6890 GC equipped with Photo Ionization Detector (PID) and Flame Ionization Detector (FID) connected in series.	Chromatographic separations for all the chemicals were obtained by GS-Q Column. The GC oven was set at 40°C for 2 min, heated at 25°C/min to 160°C and 15°C/min to 220°C and kept at 220°C for 1 min.
Gandhi, et al., 2002	HP - 5890 GC with ECD was used for TCE analysis.	Chromatographic separation was achieved on a DB-5 capillary column. The column temperature was set at 70°C with nitrogen as carrier gas at a flow rate of 2 mL/min. Detection limit of 1 µg/L was obtained for TCE.
Yanru et al., 1998	PCE, TCE, cis-DCE and VC were analyzed with a Fractovap 2900 series GC with PID. Methane and Ethane were analyzed with 5730A GC with FID.	The PID was isothermally operated at 40°C and the FID was operated at 90°C.
Hopkins et al., 1993	An automated Gas Chromatography (GC) with electron capture and hall conductivity detector was used for field measurement of TCE. The minimum TCE detection limit was 0.5 µg/L	Not available
Brown, et al., 2003	HP - 5890 Series II gas chromatograph interfaced with Micromass AutoSpec Magnetic Sector Mass Spectrometer	The electron energy and electron ionization source was set at 70 eV and the resolution was set at 1500. Chromatographic separation was achieved on DB-5ms capillary column (30 m* 0.25 mm * 0.25 µm

	was used for analysis of TCE.	film thickness). The temperature program was isothermal heating at 35°C for 4 minutes with helium as carrier gas and the retention time for TCE was 3.5 minutes.

3 Material and Methods

3.1 Experimental Approach

The suitability of PVA beads as a permeable reactive barrier in groundwater treatment was evaluated. PVA beads were made according to the method described in Thompson (1996). The physical properties of beads were characterized by conducting consolidation, permeability and density tests. The diffusion and adsorption of contaminants of interest TCE and c-DCE were evaluated by running batch experiments.

3.2 Chemicals

TCE, reagent grade (Fisher Scientific) and c-DCE, 97 % pure (Fisher Scientific), were used for analytical standards. Methanol, reagent grade (Pharmco), was used in making the chlorinated stock solutions. Pentane, HPLC grade (Fisher Scientific), and hexane, glass purified-glass distilled (Pharmco), were used as solvents for extraction of c-DCE and TCE, respectively. Boric acid, reagent grade (Fisher Scientific), poly - vinyl alcohol, molecular weight 88,000 (Fisher Scientific), calcium chloride, reagent grade (Fisher Scientific), and alginate acid (Sigma), were used in the preparation of beads.

3.3 Analytical Methods

TCE and c-DCE were analyzed using a Hewlett Packard 5890 Gas Chromatograph (GC) equipped with electron capture detector and DB-624 capillary column of 60 m length, 0.32 mm internal diameter and 1.8 μM film thickness (J&W scientific). The retention time for TCE was determined by injecting headspace samples from bottles containing pure TCE and it was observed to be 15.3 minutes. Similarly the retention time for c-DCE was observed to be 10.3 minutes. The oven temperature for the analysis was 75 °C, the inlet temperature was 200 °C and the detector temperature was 250 °C. Helium at the flow rate of 1.57 mL/min was used as the carrier gas and nitrogen was used as the make up gas at a line pressure of 25 psi.

3.4 Headspace Analysis

Calibration standards were prepared in 120 mL or 160 mL glass serum bottles containing 50 mL of distilled water. Different volumes of TCE stock solution, in methanol, were injected into serum bottles. The TCE stock solution was made by adding pure TCE in methanol. The headspace and aqueous phase concentrations in the serum bottles were calculated based on Henry's law constant (25 °C) for the addition of a known mass of TCE. The serum bottles were maintained at room temperature of 23 ± 2 °C. Henry's law constant for TCE was not available at 23 °C and therefore, the Henry's law constant at 25 °C was used as an approximation. A 20 μL sample of the headspace was injected in the GC and the peak area was plotted against mass injected which was converted to headspace concentration. Based on the Henry's constant, the headspace concentration was converted to an aqueous phase concentration. The experimental bottles were prepared in the same manner as the calibration standards and therefore, the calibration equation was used for the evaluation of concentration in experimental samples.

3.5 Liquid Sample Analysis

TCE liquid samples were analyzed by extracting 0.5 mL of a liquid sample containing TCE with 5 mL of hexane, following the procedure of Harkness et al. (1999). 3 µL of hexane was injected in the gas chromatograph and a calibration curve was plotted using peak area and mass injected. Calibration standards were prepared by adding different volumes of TCE stock solution, in methanol, into 120 or 160 mL serum bottles containing water without headspace. An aliquot of 0.5 mL of the TCE containing water from these serum bottles were transferred to 10 mL test tubes (HACH COD tubes) using a 1 mL syringe and 5 mL of pure hexane was added to the test tube. The test tube was closed with a cap and was shaken three times and the hexane and water layers were allowed to separate for 5 minutes, before injecting in GC. 3 µL of the hexane layer was injected in GC to determine the peak response.

The peak area was plotted against mass injected, which was converted to an aqueous phase concentration.

c-DCE was analyzed by extracting 1.0 mL of c-DCE containing liquid with 5 mL of pentane. A 3 μL sample of the pentane containing the extracted c-DCE was injected in the GC for determination of peak response. c-DCE stock solutions were prepared by adding a known volume of pure c-DCE in a methanol solution. The c-DCE standards were prepared by adding known amount of stock solution into serum bottles containing water.

The standards were prepared in 120 mL serum bottle with zero headspace. Liquid samples were extracted into pentane in the same manner as that described for TCE. A calibration curve was prepared by plotting the peak response against the mass injected. The calibration standards and the experimental samples were prepared in the same manner and the calibration equation created was used to calculate the concentration results in experimental samples.

Triplicate standards were used for all analysis (both TCE and c-DCE) and each sample was collected from a new standard. Samples were never taken more than once from any of the standards. The obtained results were fit to a linear equation with a R^2 value of 0.96±0.03.

3.6 PVA Beads

3.6.1 Preparation of PVA Solution

PVA solution was prepared by mixing 43.7 g of poly - vinyl alcohol in 300 mL of distilled water in a 500 mL glass beaker. The water containing poly - vinyl alcohol was placed on a hot plate with stirrer (Fisher, Model A 337856) and heated to 60 ±10°C and mixed with magnetic stirrer. The temperature was maintained until the poly - vinyl alcohol dissolved completely in solution. During the heating process, the glass beaker was covered with aluminum foil at the top to minimize loss of water due to evaporation. A thermometer was placed into glass beaker to monitor the temperature. Once the poly - vinyl alcohol dissolved in the water, 30 mL of distilled water and 3.5 mL of a 2% sodium alginate solution were added and the mixture was cooled to 35 °C. The PVA solution in its dissolved state on a hot plate stirrer with the aluminum foil cover and thermometer is shown in Figure 3-1.

3.6.2 Preparation of Sodium Alginate

The 2 % sodium alginate solution was prepared by adding 0.5 g of alginate acid to 25 mL of distilled water and stirring continuously with a magnetic stirrer until the alginate acid dissolved completely in the water.

3.6.3 Preparation of Boric Acid and Calcium Chloride Solution

Two hundred and fifty (250) grams of boric acid and 20 g of calcium chloride were added to distilled water at 40 °C to form 1 L of a saturated solution. The boric acid, calcium chloride solution was cooled to room temperature while being gently mixed using a magnetic stirrer (Nuova II S 18525).

3.6.4 Formation of PVA Beads

The PVA solution was drawn through Tygon tubing (Masterflex 7016 series) using a peristaltic pump (Cole-Parmer 7016-20). The drawn solution was dropped into the boric acid, calcium chloride solution at room temperature. The size of the beads was varied by using fixtures (22 gauge needle and connectors etc.) at tip of the tube outlets. The minimum size of the beads, on the order of 1 to 2 mm, was prepared by attaching a 22 gauge needle to the outlet tip of the tubing while the maximum size of beads, 4 to 5 mm, were made by allowing the droplets to form directly from the end of tubing without any additional fixtures.

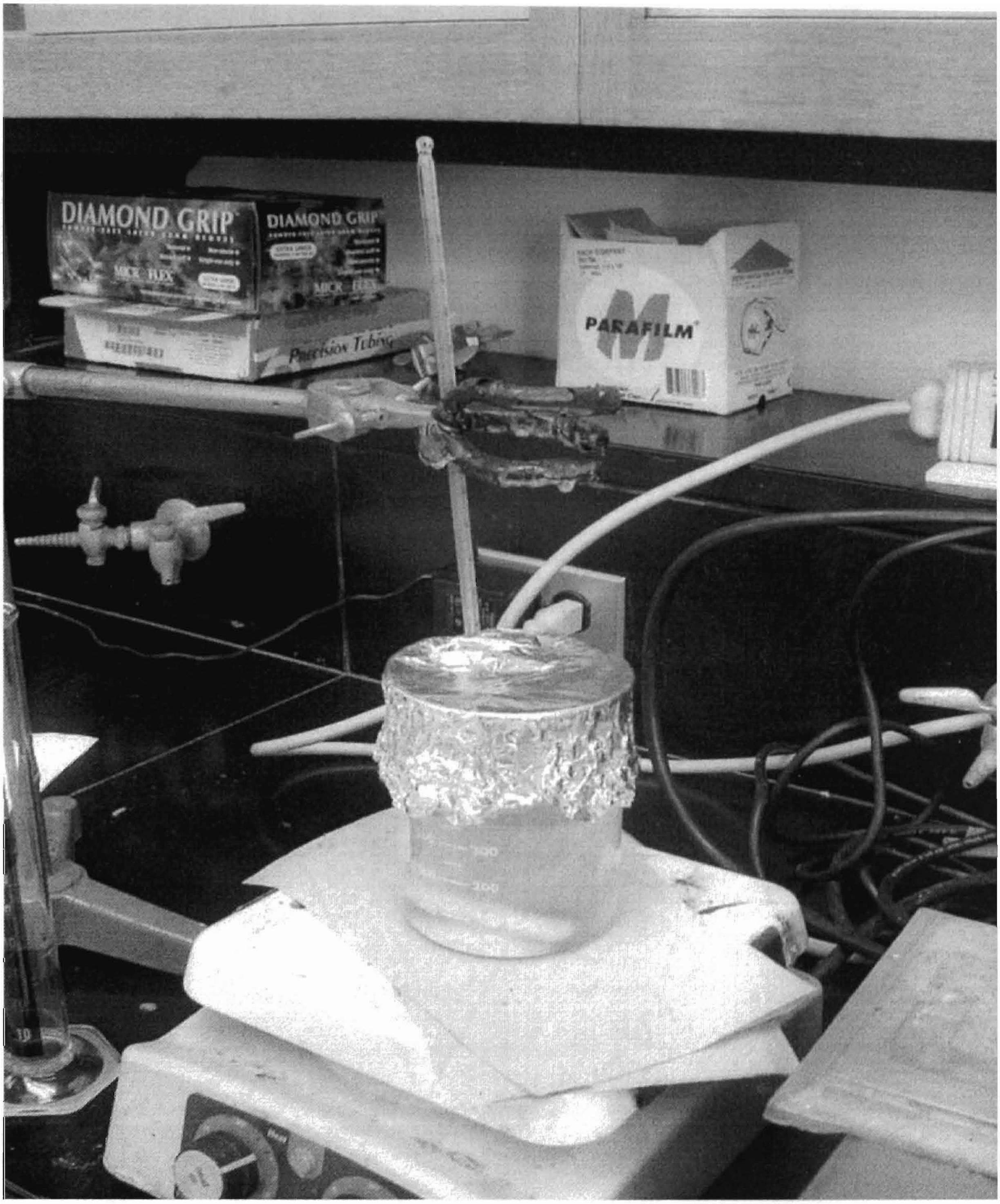

Figure 3-1 Dissolved PVA Solution on Hot Plate with Magnetic Stirrer and Thermometer

The boric acid, calcium chloride solution was gently stirred when the PVA solution was dropped into it and the PVA solution was also stirred continuously during the process. The PVA droplets were cured in the boric acid, calcium chloride solution for 24 hours. At the end of 24 hours, the beads were rinsed in distilled water and stored in glass beakers filled with distilled water. Figure 3-2 shows the PVA solution dropping into boric acid, calcium chloride solution through a fixture which was used to make beads of 3.5±0.5 mm diameter.

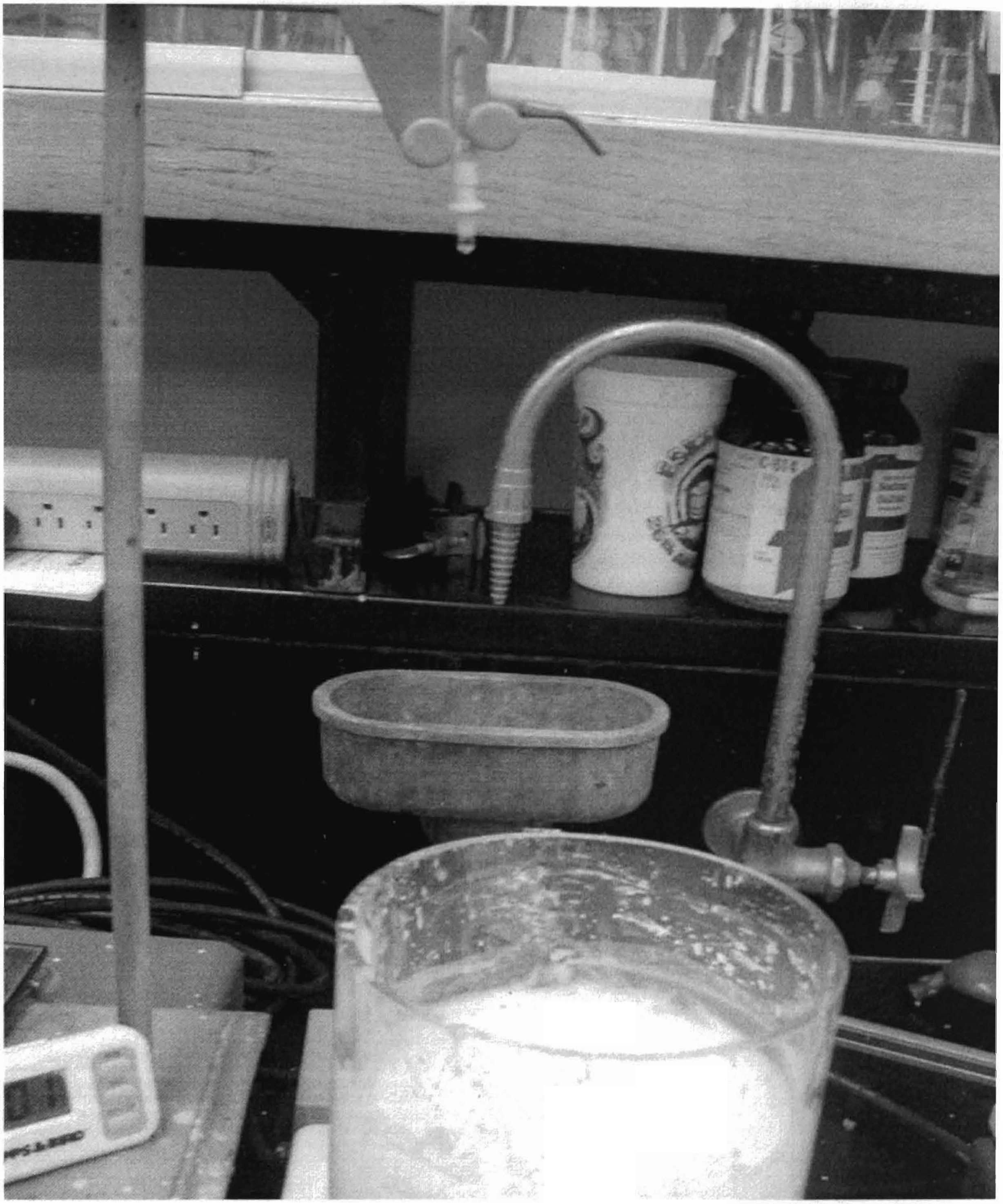

Figure 3-2 PVA Solution Falling into Boric Acid, Calcium Chloride Solution

3.7 Adsorption Test on TCE and c-DCE

The time taken for aqueous TCE to adsorb onto the surface of the PVA beads and reach an equilibrium concentration was determined by running an adsorption test. Samples were prepared in 120 mL serum bottles containing 12.5 g of PVA beads (3.5 mm diameter) with water and no headspace at room temperature (23±2 °C), and an aqueous TCE mass of 112 µg/mL. The serum bottles were closed with rubber septa and aluminum crimp caps and placed in a horizontal shaker ("Bigger Bill", Thermolyne) at 200 rpm. A 0.5 mL of liquid sample was taken from the serum bottle and extracted with 5 mL of hexane for TCE analysis. The samples were taken at 1 hour intervals during the first 7 hours and at 6 hour intervals for the next 30 hours. The last two sets of samples were taken at 102 hours and 150 hours. Triplicate bottles were used for analysis at each time period.

Similarly, the time taken by aqueous c-DCE to adsorb on the surface of PVA beads and the time taken to reach equilibrium concentration was studied. The samples were prepared with 120 mL serum bottles with water and zero headspace, and contained an aqueous c-DCE concentration of 131.72 µg/mL and 12.5 g of beads. 1.0 mL of the aqueous sample from the serum bottle was extracted with pentane for the analysis of c-DCE concentration in liquid. The adsorption study samples were placed on a horizontal shaker ("Bigger Bill", Thermolyne) and rotated at 200 rpm. In addition the bottles were shaken manually 10 minutes before the analysis so as to make sure the beads had sufficient contact with the c-DCE solution.

3.8 Coefficient of Diffusion for TCE and c-DCE

The diffusion test was done to determine coefficient of diffusion for TCE and c-DCE into the beads. The change in liquid TCE and c-DCE concentrations with respect to time was used to determine the diffusion coefficient. The diffusion coefficient was determined by the shrinking core model (SCM). The SCM is described by following equation (Chen et al., 1993):

$$1-3(1-X)^{2/3}+2(1-X)=6D(\int Cdt)/R^2C^o \tag{1}$$

$$F(X)=1-3(1-X)^{2/3}+2(1-X) \tag{2}$$

The extent of reaction (X) was determined by the following expression (Chen et al., 1993):

$$X=(C_o-C)/(C_o-C_\infty) \tag{3}$$

The average binding site density of PVA (Co) was determined by following expression (Jang, 1994):

$$Co=(Co-C\infty)*(\mathrm{R}V/VS) \tag{4}$$

The diffusion coefficient (D) was determined from the following equation (Chen et al., 1993):

$$D=(Slope)C_oR^2/6 \tag{5}$$

C = Concentration in solution at any given time (mg/L)

C^0 = Initial Concentration in solution (mg/L)

$C\infty$ = Concentration in solution at equilibrium (mg/L)

C_o = Average site binding density of PVA (mg/L)

D = Diffusion coefficient (cm^2/sec)

R = Radius of bead (cm)

T = Time (s)

X = Extent of reaction

RV = reactor volume (cm^3)

VS = Volume of spheres (cm^3)

3.9 Physical Properties of PVA Beads

The physical properties such as permeability, consolidation and density of PVA beads were determined to study the suitability of the PVA beads as a permeable reactive barrier for the treatment of contaminated groundwater. The physical properties were studied using 3.5 ±0.5 mm beads.

3.9.1 Consolidation

A consolidation test was conducted on PVA beads as per the procedure outlined in the soil mechanics laboratory manual by Das (1997). Drained beads were packed into the sample cell of an oedometer and the ability of the beads to withstand overburden pressure was evaluated. The consolidation test was done for consolidation pressures of 170, 250, 500, 1000 and 2000 lb/ft^2 and the time required for 50% consolidation and 90% consolidation were calculated for the 170 and 250 lb/ft^2 loading condition. For the other loading conditions, the consolidation was not significant enough to calculate the time required for 90% and 50% consolidation. A damp cloth was placed on top of the consolidation ring to maintain moisture and prevent the beads from drying.

3.9.2 Determination of Maximum Consolidation Pressure

The consolidation test was conducted to determine the maximum consolidation pressure that the PVA beads can withstand, and this value was converted to soil depth. The initial consolidation pressure was set at 250 lb/ft^2 and the consolidation pressure was increased at 30 minutes intervals and the maximum consolidation pressure used in the experiment was 16,000 lb/ft^2. The test was conducted for 150 minutes.

The density of the soil was assumed as 100 lb/ft^3 (Snethen, 2002) and this value was used to convert the consolidation pressure to depth of soil. Based on the assumed density, the beads were subjected to soil depth of 2.5 ft to 160 ft. The consolidation ring was filled with water to keep the beads moist and prevent them from drying. Figure 3-3 shows the oedometer experimental set up for the consolidation test, and Figure 3-4 shows the consolidation test with a damp cloth covering the beads to maintain the moisture.

3.9.3 Permeability

Permeability test was conducted on PVA beads of 3.5 ± 0.5 mm size, as per the procedure outlined in the soil mechanics laboratory manual by Das (1997) using constant head permeability apparatus. The permeability test was carried out to determine the coefficient of permeability by a constant-head method with laminar flow of water through consolidated beads.

Figure 3-5 shows the experimental set up for constant head permeability test. Drained beads were packed in the sample cylinder of the constant head permeability apparatus. Geomembrane filter screens were placed at the top and bottom of the sample cylinder. The liquid flowing out of the sample cylinder was collected in a graduated cylinder and the corresponding initial and final time was recorded.

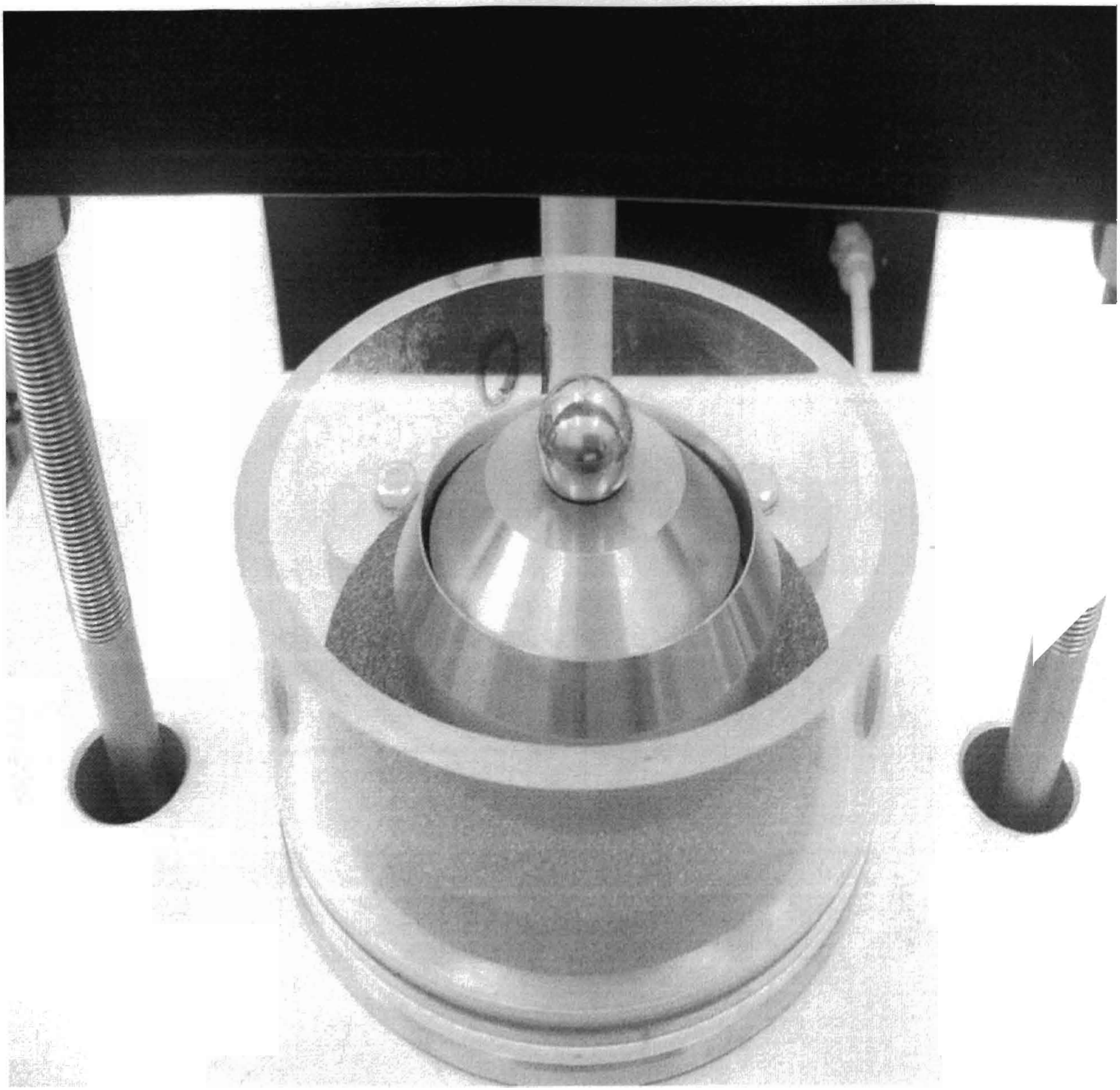

Figure 3-3. Consolidation Test Apparatus

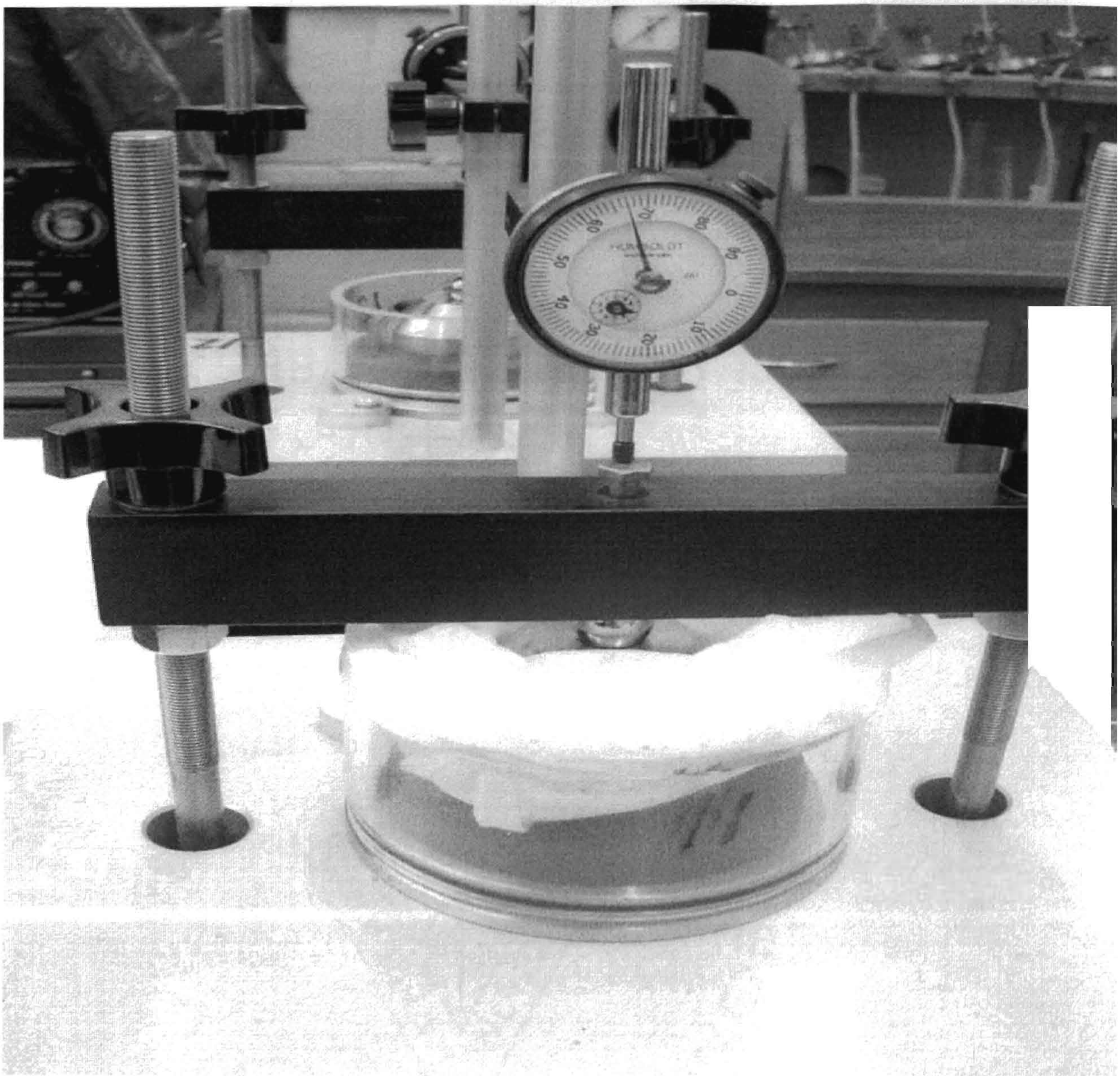

Figure 3-4. Consolidation Test with a Damp Cloth Covering

Figure 3-6 shows the beads after being subjected to the permeability test and the sample cylinder of a constant head permeability apparatus. The coefficient of permeability was determined by the expression from Das (1997):

$$k = Q * L / (A * h * t) \tag{6}$$

k = Coefficient of permeability

Q = Volume of water collected

A = Sample cross sectional area

H = head

T = Sample collection time.

3.9.4 Coefficient of Permeability of Consolidated Beads

To determine the coefficient of permeability for consolidated beads, the beads were consolidated at a given overburden pressure and tested for permeability. When beads are used as a permeable barrier in groundwater remediation, the beads have to support the weight of the soil layer above them. Therefore, the beads were subjected to overburden pressures equivalent to the soil depth of 5 ft, 10 ft and 20 ft.

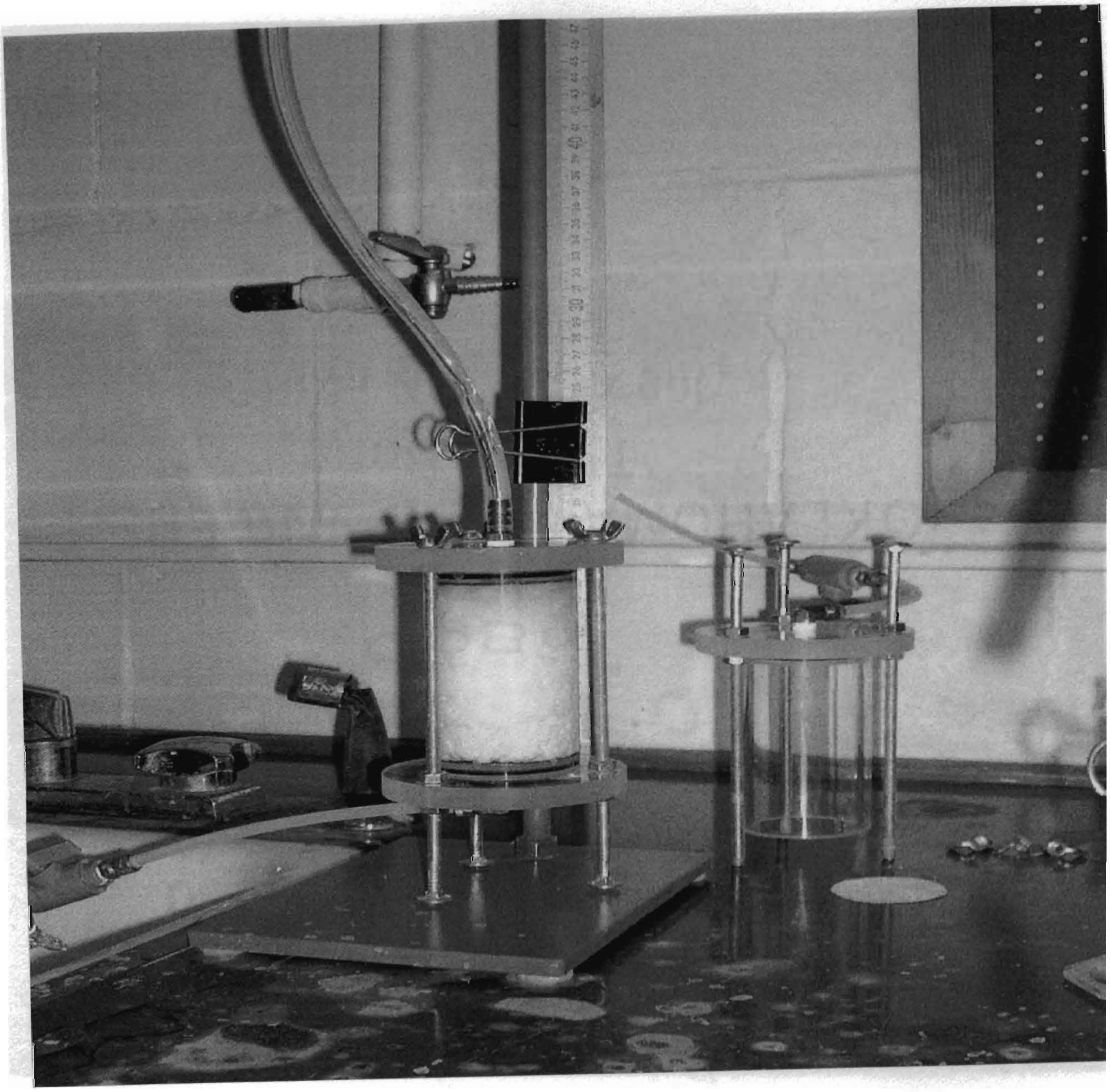

Figure 3-5. Permeability Test Apparatus

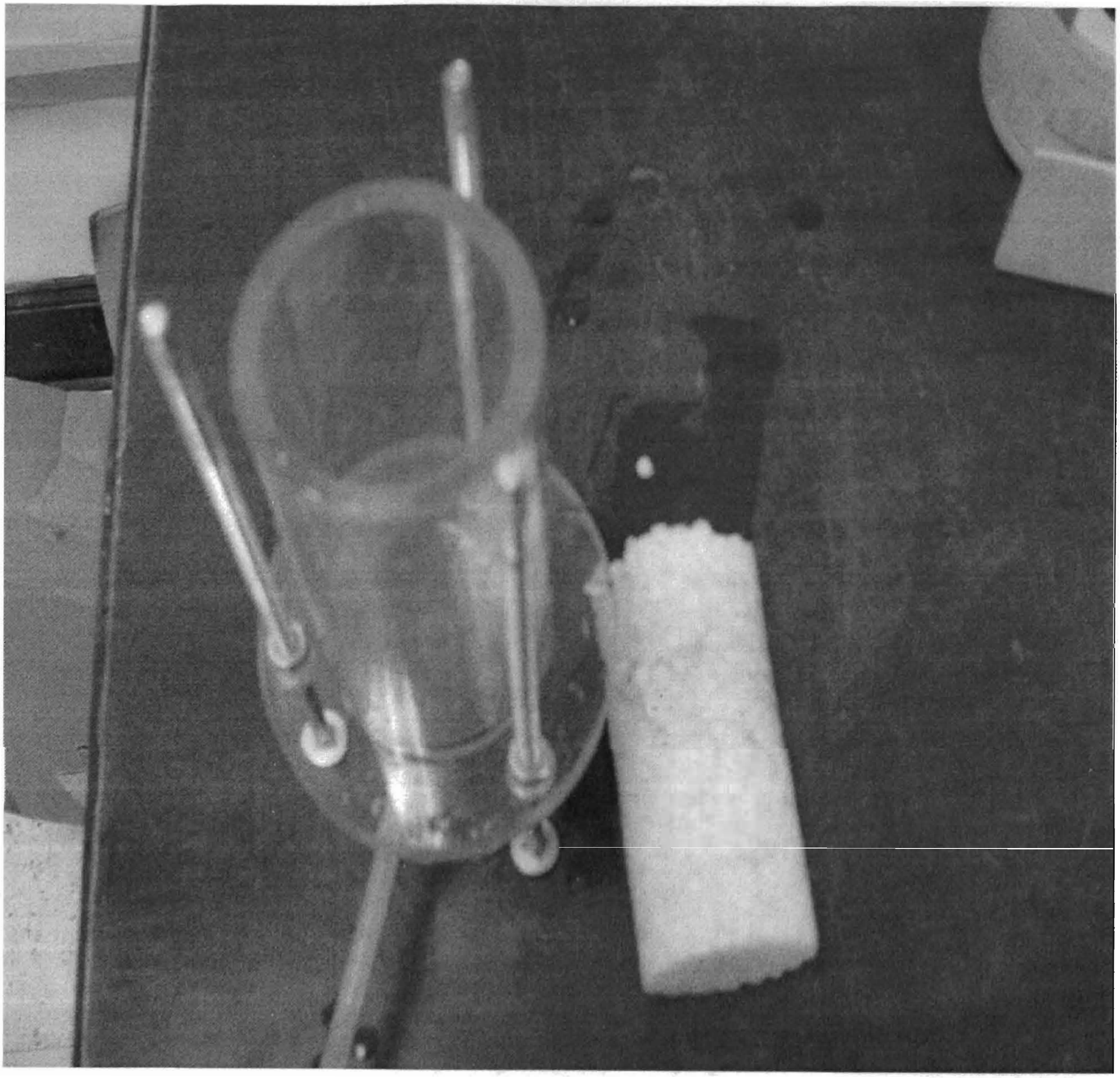

Figure 3-6. Beads after Permeability Test

To simulate the overburden pressure due to 5 ft soil, the beads were subjected to a consolidation pressure of 500 lb/ft^2. The consolidation experiment was conducted on an oedometer, in the OSU Soil Mechanics Laboratory, with beads of 3.5 ±0.5 mm diameter. The consolidated samples from these four instruments were used to pack the sample cylinder of constant head permeability apparatus. The four layers of beads were filled inside the cylinder of the constant head permeability apparatus. Figure 3-7 shows the shape of the beads consolidated at 500 lb/ft^2, before placement inside the sample cylinder of the constant head permeability apparatus. Similarly, two more sets of fresh beads of the same size were consolidated at overburden pressures of 1000 lb/ft^2 and 2000 lb/ft^2 and corresponding permeability values were measured.

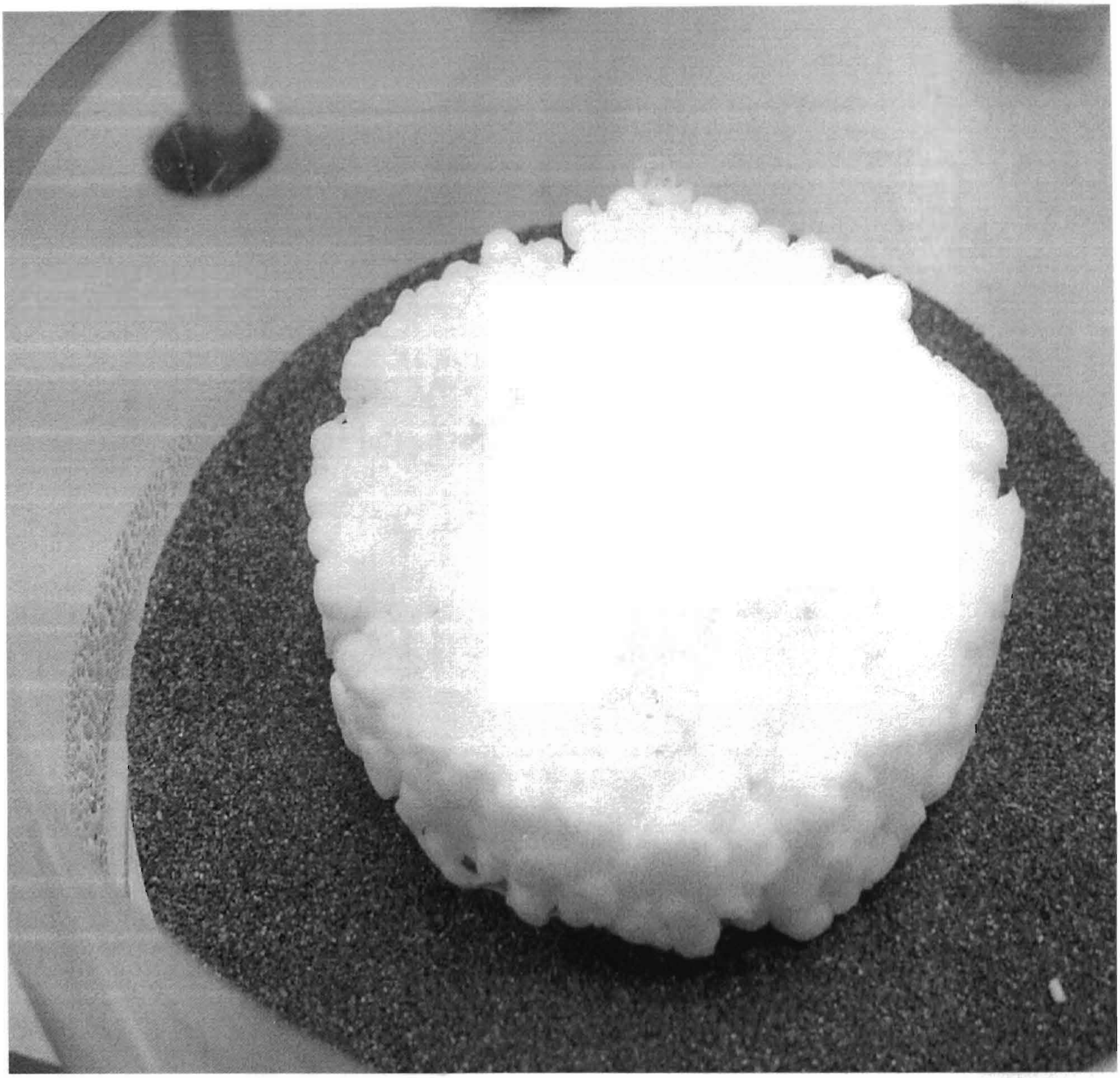

Figure 3-7. Shape of Beads Pre-Consolidated at 500 lb/ft^2, before Testing for Permeability

3.9.5 Density

The density of beads was calculated for two different sizes of beads, 2 mm diameter and 4 mm diameter and also for one set of transparent beads. The beads that were used for the adsorption study turned transparent during the course of the experiment. The reason for the beads turning transparent was not known. The transparent beads and the normal white solid beads are shown in Figure 3-8.

These beads were collected in a zip lock bag and stored in a refrigerator at 39°F and used for the determination of density, to check if they were different from the original form (solid white) of the beads. The density was calculated for three different mass of beads 5 g, 10 g and 15 g. For each mass of beads, the density was measured in a graduated cylinder with an initial volume of 30 mL, 40 mL and 50 mL. A known mass of beads was added to the initial volume of water and the final volume of water was recorded. The density at room temperature (22°C±2) was calculated as the mass of beads divided by the change in volume of water.

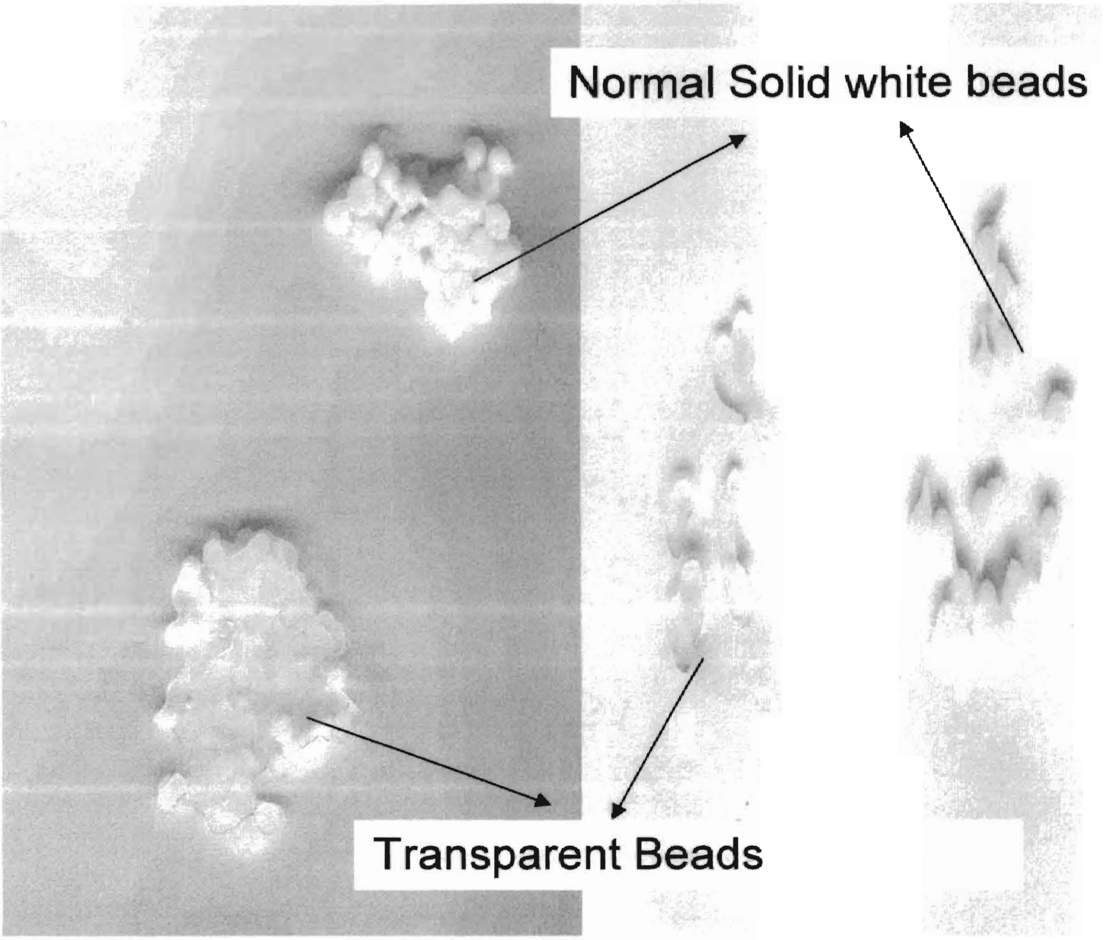

Figure 3-8 Normal White Solid Beads and Transparent Beads

4 Results and Discussion

Experiments were conducted to study the feasibility of using PVA beads as a permeable reactive barrier for groundwater remediation. Physical properties of the beads such as consolidation pressure, permeability, soil overburden pressure and density were determined. An adsorption studies were conducted for both TCE and c-DCE to determine the physical removal of the contaminants by the beads.

4.1 PVA Bead Stability

The PVA beads were prepared as discussed in the materials and methods section. The beads were spherical in shape and remained intact over the period of 1 year. The beads that were prepared on 10/14/02 and cured in distilled water retained their shape and size when observed on 11/18/03. Even after 1 year when stored in distilled water the beads did not dissolve. Loss of water was observed in the beakers in which beads were stored. Distilled water was added at random intervals to the beads so as to prevent drying of beads. Therefore, the beads were always stored in distilled water during the one year observation period. An interesting observation was that beads, when washed with tap water, turned transparent. The reason for this change in color is unknown.

4.2 Physical Characteristics of Beads

The physical properties such as consolidation pressures, permeability and density were determined for PVA beads. The consolidation pressure was converted in terms of soil depth or overburden pressure that the beads can withstand when placed in field conditions. The permeability test was conducted to determine the suitability of PVA beads for groundwater applications where the barrier permeability should be close to the groundwater permeability, so as not to alter the existing ground water hydrology.

4.2.1 Consolidation Test

The coefficient of consolidation was determined using consolidation test. The coefficient of consolidation was evaluated based on the time taken for 90% consolidation, difference in sample height before and after consolidation and time factor of 0.848 (constant from Das, 1997). The square root of time fitting method as described in Venkatramaiah (1993) was used in evaluation of coefficient of consolidation from the oedometer results. The time required for 50 % (t_{50}) and 90 % (t_{90}) consolidation was determined from the plot of consolidation dial gauge reading versus square root of the time. The t_{50} and t_{90} values were found for consolidation loads of 170 lb/ft^2 and 250 lb/ft^2. For higher loads such as 500, 1000 and 2000 lb/ft^2, the consolidation was rapid and it was not possible to collect data for the calculation of t_{50} and t_{90} but a separate test was conducted under these loading conditions to calculate the permeability.

The t_{50} and t_{90} for 170 and 250 lb/ft^2 are shown in Table 2.0. Figure 4-1 and Figure 4-2 show the consolidation curves for 170 lb/ft^2 and 250 lb/ft^2, respectively. For the consolidation pressures of 170 lb/ft^2 and 250 lb/ft^2, the primary consolidation was reached within 450 minutes, while beyond that time a slow secondary consolidation phase occurred. The primary consolidation was more rapid at 250 lb/ft^2 compared to the 170 lb/ft^2 and the secondary consolidation was minimal at 250 lb/ft^2 compared to 170 lb/ft^2. For a loading pressure of 170 lb/ft^2, the percentage consolidation after 450 minutes was 9 % where as the percentage consolidation after 450 minutes at 250 lb/ft^2 loading was 0.2 %.

Figure 4-1 and Figure 4-2 are the plots for square root of time and therefore the time taken for the primary consolidation of 450 minutes is numerically represented as 21.21 in Figure 4-1. According to Venkatramaiah (1993) the time settlement curve has three phases, the elastic phase, primary consolidation phase and the secondary consolidation phase. The instantaneous elastic compression is due to the dissipation of excess hydrostatic pressure and the primary consolidation is due to the decrease in void ratio in the material, which is a slow process (Venkatramaiah, 1993). The secondary consolidation is defined as the phenomenon of continued consolidation beyond complete dissipation of excess pore water pressure of the primary consolidation and it can be 20 % or more (Venkatramaiah, 1993).

Figure 4-1 and Figure 4-2 do not clearly establish the elastic compression of the time settlement curve. The elastic compression is instantaneous and it can fit in the first few minutes of the time settlement curve. Moreover, the tested beads are not perfectly cohesive and hence distinguishing a clear elastic compression was found to be a difficult task.

The coefficient of consolidation values are shown in Table 2.0. The coefficient of consolidation is one of the important parameters used in evaluating the amount of consolidation that the beads can undergo under a given overburden pressure or the pressure due soil on top of the beads. From Table 2, it can be observed that as the applied pressure increases, the time taken for consolidation decreases and the consolidation coefficient increases. Therefore, the increasing pressure will cause faster consolidation. According to Venkatramaiah (1993), the consolidation coefficient has a wide range from $5*10^{-4}$ mm^2/sec to $2*10^{-2}$ mm^2/sec. Therefore, coefficient of consolidation of PVA beads fits into the general range.

Table 2. Time for 50 % and 90 % Consolidation

Consolidation Load (lb/ft^2)	T_{50}(minutes)	t_{90}(minutes)	Coefficient of Consolidation (C_v) mm^2/sec
170	14	165	4.82E-3
250	10	55	1.45E-2

The consolidation test estimates the settlement of beads at a given loading condition. The behavior of beads at varying loading conditions was tested by a compressibility test. The compressibility test measures the maximum strain in beads for increasing loading conditions. Figure 4-3 shows the compressibility test results for the beads at various pressures. The beads under increasing load reached 56% cumulative strain at an overburden pressure of 4000 lb/ft^2.

For a soil with density of 100 lb/ft^3, 4000 lb/ft^2 would correspond to a depth of 40 ft. Initial stress response up to 4000 lb/ft^2 was rapid as, illustrated in Figure 4-3, which shows a steep increase in cumulative strain. Loads higher than 4000 lb/ft^2 did not produce appreciable strain on the beads beyond 56%. The beads were in elastic range up to 4000 lb/ft^2 load and beyond 4000 lb/ft^2, the beads reached the plastic range. Under elastic range, the beads were able form the original shape after removing the load and in the plastic range the beads did not come back to the original shape after the removing the load. The breaking point or the breaking load at which the beads completely collapse was not reached at 16,000 lb/ft^2. The beads were subjected to maximum pressure of 16,000 lb/ft^2 which would correspond to 160 ft in terms of soil depth, for a soil with density of 100 lb/ft^3. Therefore, significant amount of compression occurs at shallow depth at faster rate and decreases with increasing depth.

The consolidation results are shown in Table 3 and Table 4 while the compressibility results are shown in Table 5. Thompson (1996) studied the compression behavior of PVA beads and found the maximum of cumulative strain as 48 % for 4000 lb/ft^2.

Table 3 Consolidation Results at 170 lb/ft^2

Time (min)	Dial gauge reading	(Time) $^{0.5}$
0.1	0.755	0.32
0.25	0.743	0.50
0.5	0.736	0.71
1	0.729	1.00
2	0.72	1.41
4	0.708	2.00
8	0.692	2.83
15	0.676	3.87
30	0.663	5.48
60	0.642	7.75
120	0.635	10.95
390	0.605	19.75
450	0.605	21.21
510	0.6	22.58
570	0.6	23.87

Table 4 Consolidation Results at 250 lb/ft^2

Time (min)	Dial gauge reading	(Time) $^{0.5}$
0.1	0.564	0.32
0.25	0.56	0.50
0.5	0.56	0.71
1	0.559	1.00
2	0.556	1.41
3	0.554	1.73
4	0.552	2.00
5	0.551	2.24
6	0.548	2.45
7	0.547	2.65
8	0.546	2.83
9	0.545	3.00
10	0.544	3.16
20	0.54	4.47
30	0.536	5.48
40	0.531	6.32
50	0.53	7.07
60	0.528	7.75
120	0.526	10.95
1560	0.526	39.50
3000	0.526	54.77
3960	0.525	62.93

Table 5 Compressibility Results

Load on Specimen (PSF)	Dial gauge reading	Time (min)	Cumulative Strain (%)
250	0.8	0	0.00
250	0.55	30	31.25
500	0.53	30	33.75
500	0.458	60	42.75
1000	0.447	60	44.13
1000	0.4	90	50.00
2000	0.391	90	51.13
2000	0.362	120	54.75
4000	0.36	120	55.00
4000	0.352	150	56.00
8000	0.351	150	56.13
8000	0.351	180	56.13
16000	0.35	180	56.25
16000	0.35	210	56.25

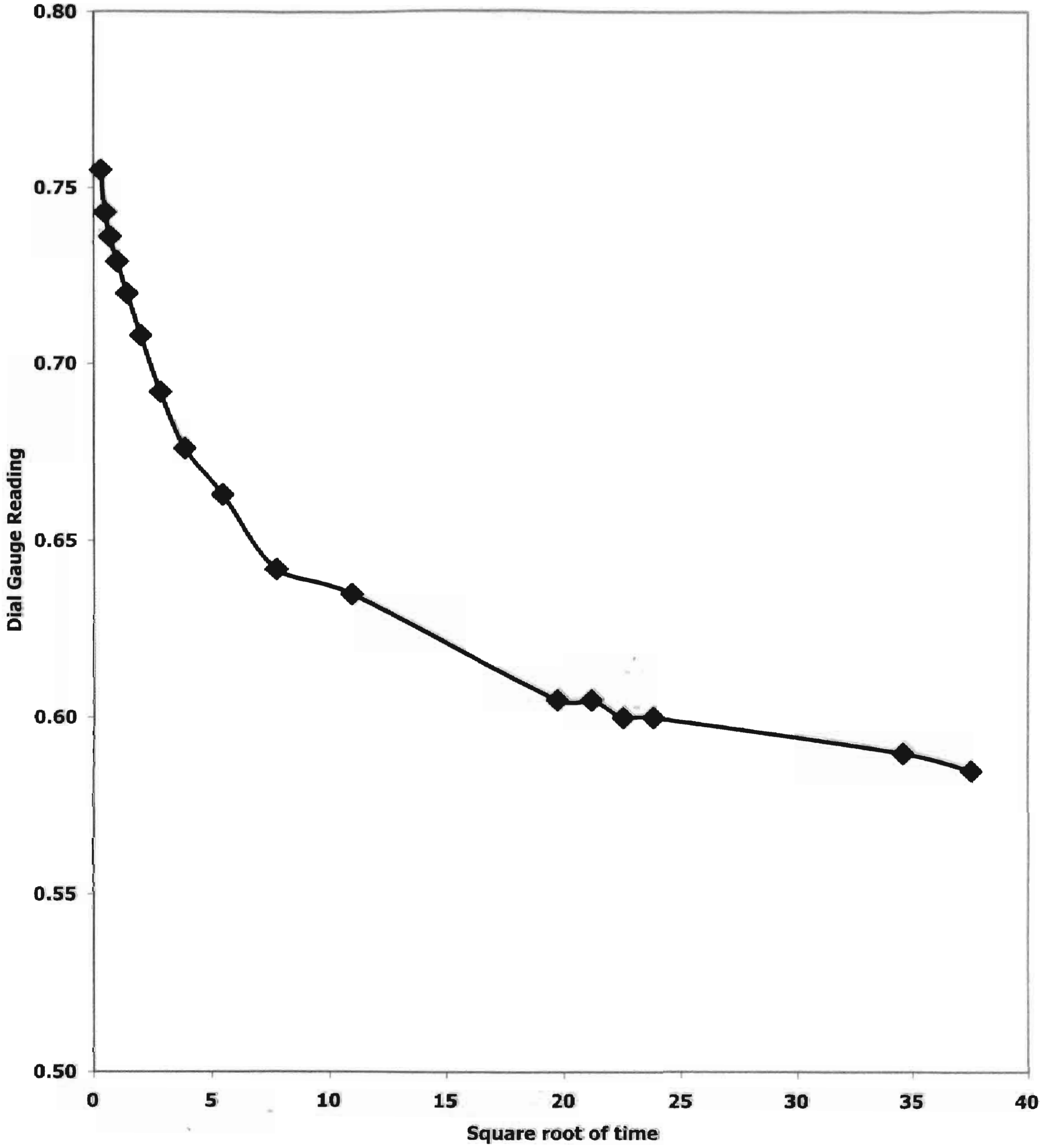

Figure 4-1 Consolidation Curve at 170 lb/ft^2

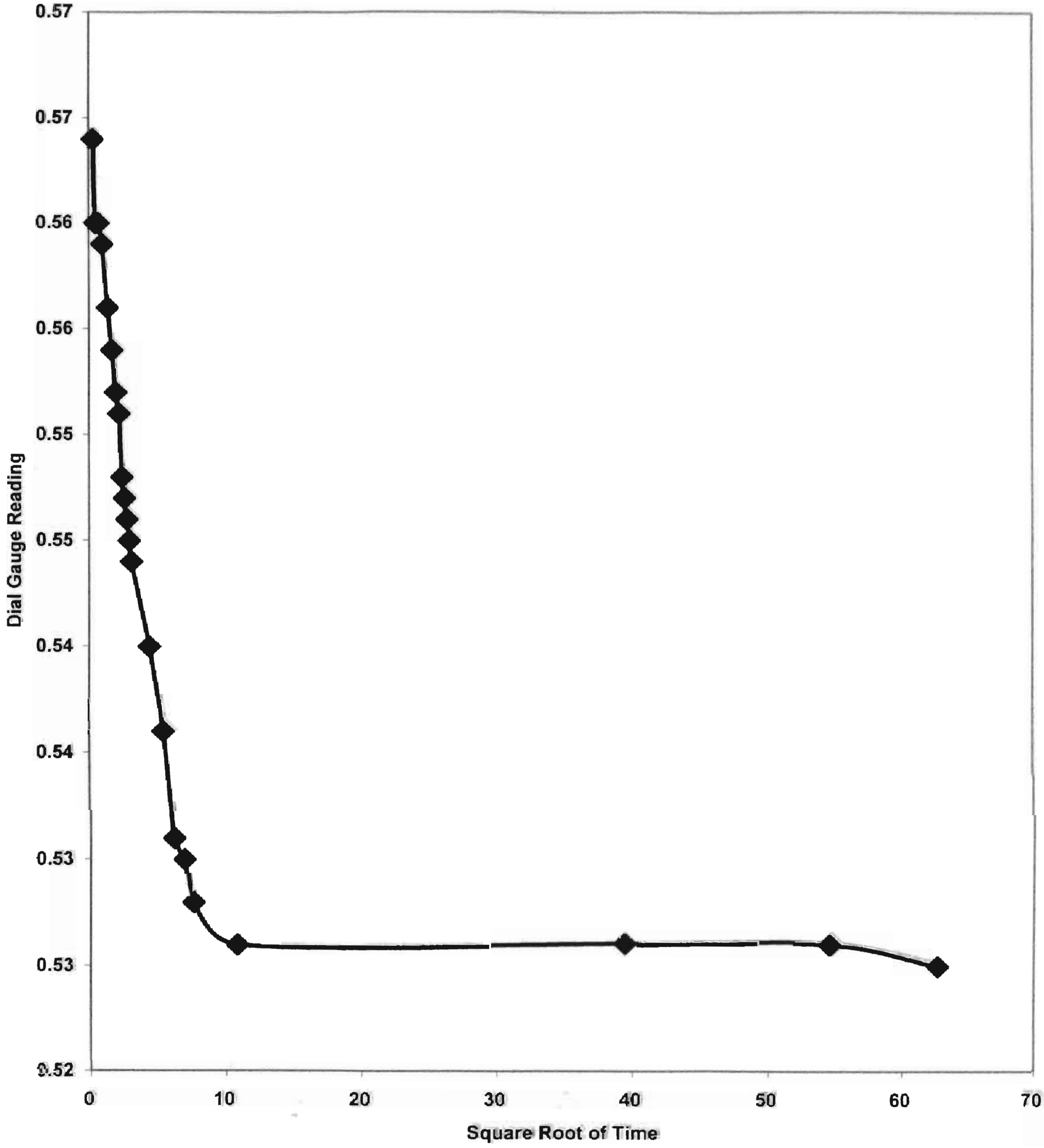

Figure 4-2 Consolidation Curve at 250 lb/ft^2

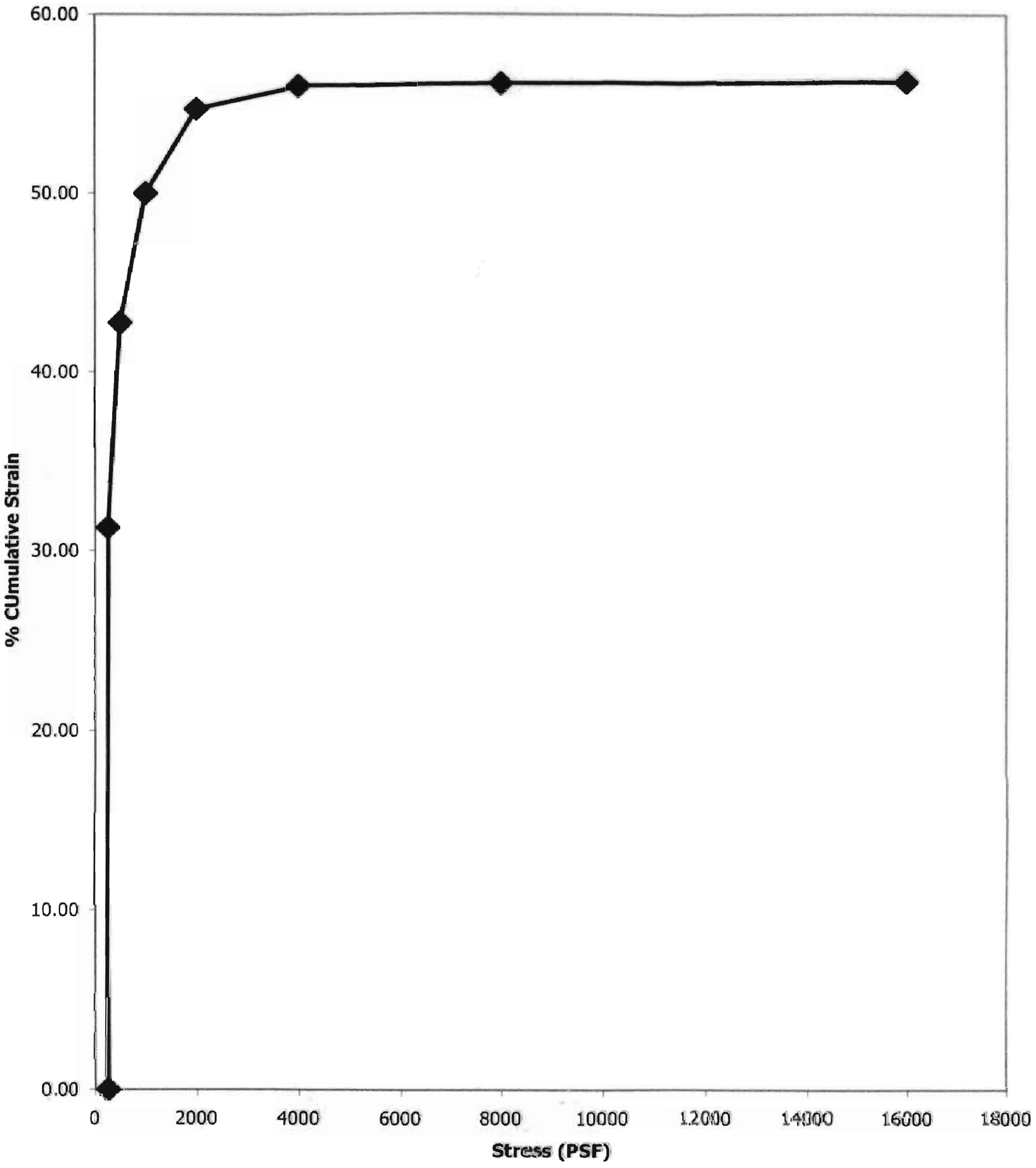

Figure 4-3 Compressibility of PVA Beads

4.2.2 Permeability Test

Permeability is defined as the ease with which fluid flows through the soil or material under study and it depends on characteristics of the fluid and the permeable material (Venkatramaiah, 1993). A constant head permeability test was conducted on pre - consolidated beads to determine the coefficient of permeability of the PVA beads. The constant head permeability test works on the principle of maintaining a constant hydraulic head and measuring the quantity of fluid flowing through the material at a given time (Smith 1982).

The permeability values of beads at consolidation pressures of 500, 1000 and 2000 lb/ft^2 are shown in Figures 4-4, 4-5 and 4-6, respectively. The figures were made by plotting permeability at a given consolidation pressure and the time at which permeability was measured.

Figure 4-4 shows the permeability value for the beads subjected to a consolidation pressure of 500 lb/ft^2. Water was allowed to flow through the beads with a constant hydraulic head of 80 cm and the time required for collection of 300 mL of water was recorded. The hydraulic head of 80 cm was arbitrarily fixed for the practical convenience of running the experiment. The 80 cm hydraulic head was convenient to keep the hydraulic head constant by continuously adding water and at the same time collecting water through the outlet of the apparatus. The volume of water collected (300 mL) was also arbitrarily fixed so as to have sufficient time to run the experiment. The water samples were collected on three consecutive days. For the first two days 300 mL of water was collected four times and the corresponding permeability was calculated.

The permeability values and the corresponding times taken to collect 300 mL sample are shown in Table 6 and are graphically shown in Figure 4-4. From Table 7 it can also be observed that there is gradual drop in permeability. The time taken to collect first 300 mL of water was 35 seconds and the time taken for the collection 300 mL water for the fourth time was 59 seconds. As the water flows across the bead bed, the beads tend to realign and fill the void spaces in between them and thereby decrease the permeability.

In order to check the variation in permeability with respect to time, the water flow across the bead was stopped and the head was maintained constant at 80 cm. Permeability measurements were made again after 24 hours and the results are shown in Table 7 and graphically presented in Figure 4-4. The permeability values in Figure 4-4 are the average of 4 samples. The average permeability on day 1 varied by 8 % with respect to the average permeability on day 0. As seen on day 0, the permeability value decreased as the volume of water collected increased, which supports the concept that the flowing water realigns the beads to fill the void spaces between them.

The water flow was stopped and the beads were allowed to be under 80 cm hydraulic head for three more days. Permeability measurements were done at the end of four days, and the results are shown in Table 8. For permeability measurement on day four, 8 different samples were collected. For the first six measurement 300 mL of water was collected and for the last two measurements 600 mL and 1000 mL water was collected, respectively. The average permeability on day 4 varied by 50 % compared to day 0 and compared to day 1, the permeability on day 4 varied by 45 %. As observed on day 0 and day 1, the permeability values on day 4 decreased.

The decrease in permeability over a given period time may be due to the fact that as water flows through the beads, it can knocks out tiny particles from their position and clog the filters at the bottom and also the flowing water can move the beads and make the bead bed more compact.

Permeability measurements were done for a pre-consolidation pressure of 1000 lb/ft^2, which could be equivalent to the pressure exerted by 10 ft of soil over the beads. The permeability measurements for the beads consolidated at 1000 lb/ft^2 was done on day 0 and day 1 (at the end of 24 hours) and the results are shown in Table 9 and Table 10 respectively. Figure 4-5 shows the permeability results for day 0 and day 1 for 1000 lb/ft^2. The permeability values in Figure 4-5 are the average of 5 samples. The permeability results for the beads consolidated at

1000 lb/ft^2 was stopped at the end of 24 hours, based on the suggestion by Snethen, (2003), that the typical permeability measurements are not done beyond 24 hours. The hydraulic head was maintained at 80 cm during the 24 hour period.

Table 6 Day 0 Permeability Results at 500 lb/ft^2

Volume of water (mL)	Time (Sec)	Permeability (cm/sec)
300	35	0.053
300	45	0.041
300	53	0.035
300	59	0.031

Table 7 Day 1 Permeability Results at 500 lb/ft^2

Volume of water (mL)	Time (Sec)	Permeability (cm/sec)
300	45	0.041
300	47	0.039
300	54	0.034
300	57	0.032

Table 8 Day 4 Permeability Results at 500 lb/ft^2

Volume of water (mL)	Time (Sec)	Permeability (cm/sec)
300	62	0.030
300	72	0.026
300	81	0.023
300	90	0.021
300	100	0.019
300	105	0.018
600	260	0.014
1000	557	0.011

The permeability values on day 0 varied by 29 % among the five measured values. The permeability values on day 1 varied by 32 % among the five measured values. The variation between the average permeability value on day 0 and day 1 was 50 %. The variation in permeability of the beads subjected to consolidation pressure of 1000 lb/ft^2 was consistent with the variation observed in the permeability values of beads consolidated at 500 lb/ft^2. The average permeability of the beads consolidated at 1000 lb/ft^2 varied by 3 % compared to the average permeability of the beads consolidated at 500 lb/ft^2.

Permeability measurements were also done for the beads consolidated at 2000 lb/ft^2, which would be equivalent to the pressure exerted by soil of 20 ft depth. Table 11 and Figure 4-6 show the permeability results of beads consolidated at 2000 lb/ft^2. The beads that were consolidated at 2000 lb/ft^2 settled faster in the permeability apparatus with flowing water.

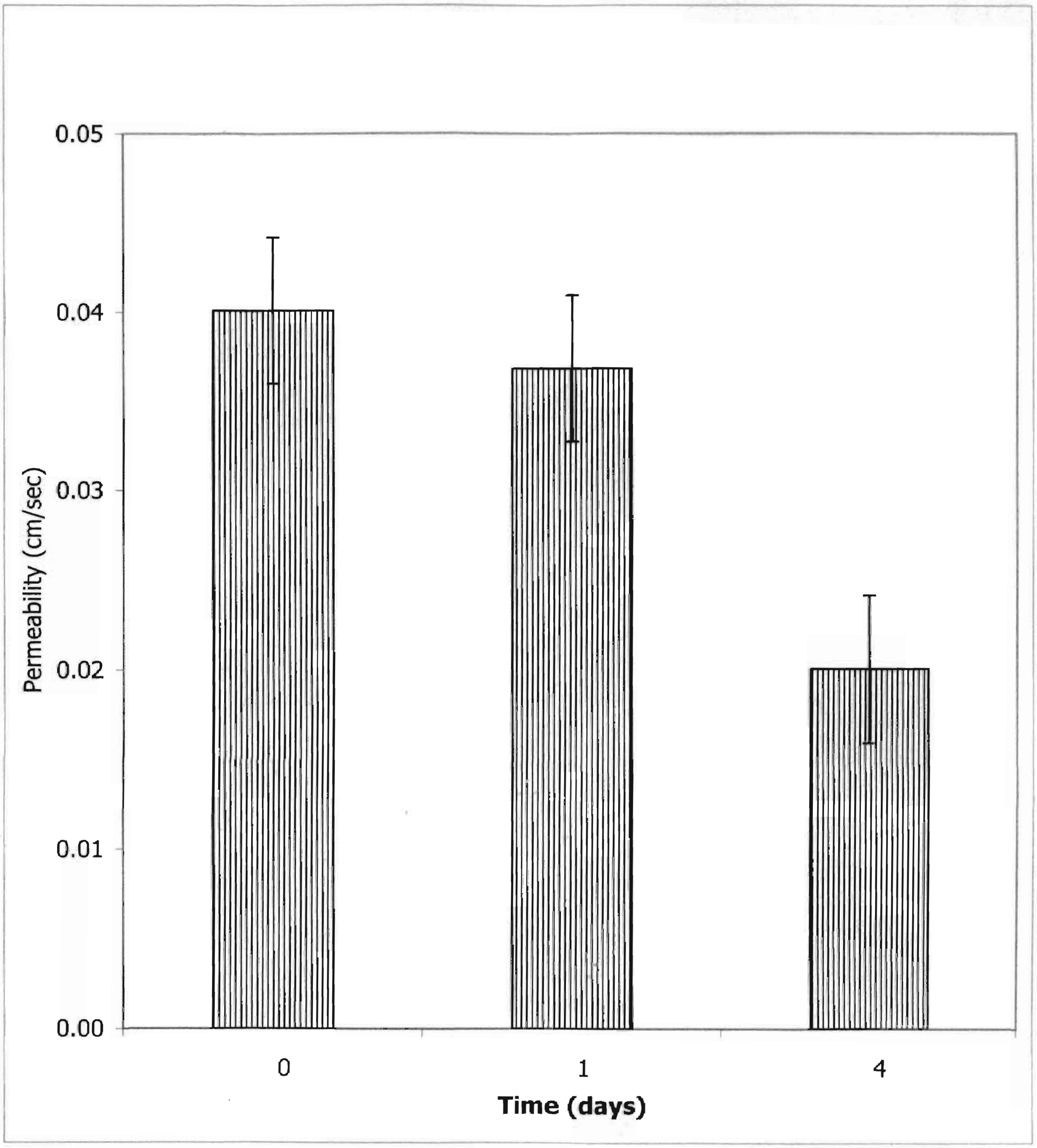

Figure 4-4 Permeability at Pre-consolidation Pressure of 500 lb/ft^2

The permeability values were very low and the sample collection time was also high. The results shown in Figure 4-6 are for the data collected within 8 hours from the starting time of the experiment. A large drop in permeability was observed within the 8 hours. 96 % variation was observed between the permeability values measured at the beginning of the experiment and at the end of 8 hours. The sample was continuously collected during the 8 hour time period. The continuous flow of water can increase the settlement of beads within the permeability apparatus and decrease the flow.

Table 9 Day 0 Permeability Results at 1000 lb/ft^2

Volume of water (mL)	Time (sec)	Permeability (cm/sec)
300	28.39	0.065
300	38.32	0.048
300	34	0.054
300	39	0.047
300	40	0.046

Table 10 Day 1 Permeability Results at 1000 lb/ft^2

Volume of water (mL)	Time (sec)	Permeability (cm/sec)
300	58	0.032
300	65	0.028
300	70	0.026
300	80	0.023
300	86	0.022

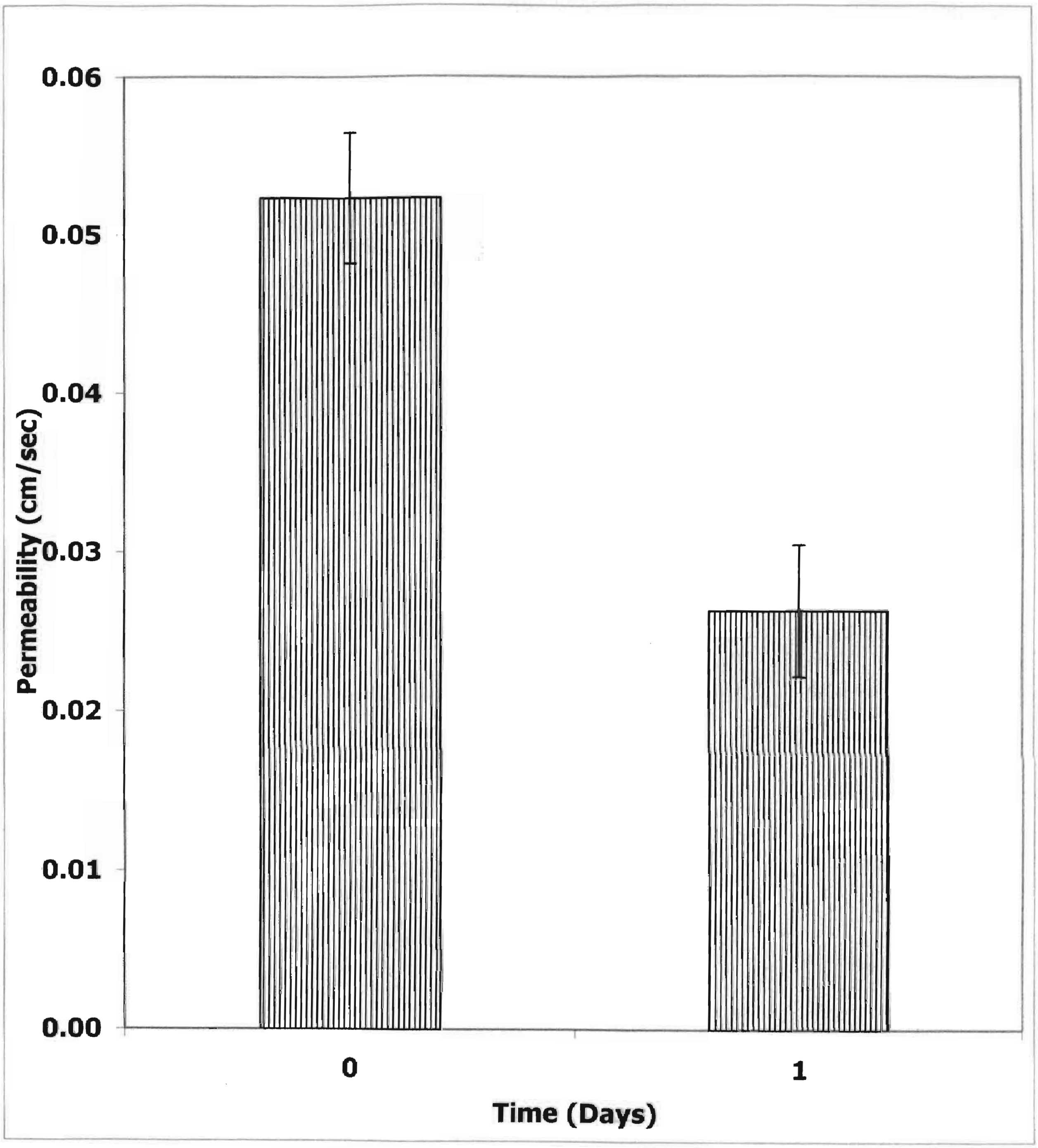

Figure 4-5 Permeability at Pre-consolidation Pressure of 1000 lb/ft^2

For the consolidation pressure of 500 and 1000 lb/ft^2, the sample collection time for 300 mL of water was less than a minute for the first sample and it increased gradually for subsequent set of samples. For 2000 lb/ft^2 consolidation pressure, the sample collection time for the first 300 mL of water was 60 minutes, which was much higher than the time required for the same observation under low consolidation pressures. Since, sample collection time increased, continuous flow of water was maintained during the 8 hour period.

For the lower consolidation pressure, collection time varied from less than a minute to 10 minutes. Therefore, the water flow value was closed and reopened later to collect samples. The permeability values for the beads consolidated at 2000 lb/ft^2 was calculated for different volumes of water collected and corresponding time but the permeability measurements for the beads consolidated at 500 lb/ft^2 and 1000 lb/ft^2 were measured for a constant volume of 300 mL.

The average permeability varied from 0.04 cm/sec at 500 lb/ft^2 to 3.74E-4 cm/sec for the 2000 lb/ft^2. According to McGovern et al. (2002), the subsurface hydraulic conductivity can vary between 2.08E-3 to 1.89E-10 cm/sec. The permeability values measured for the beads at the maximum consolidation pressure of 2000 lb/ft^2 fall within the range reported by McGovern et al. (2002). The hydraulic conductivity is also site specific and the application of beads as permeable barrier could depend on the permeability of the site and the depth of soil layer over the beads.

Table 11 Permeability Results at 2000 lb/ft^2

Volume of water (mL)	Time (Sec)	Permeability (cm/sec)
300	1320	1.4E-03
145	2880	3.1E-04
45	6000	4.6E-05
50	8400	3.7E-05
75	8400	5.5E-05

The relationship between depth soil with 100 lb/ft^3 density and the permeability are shown Table 13 and graphically presented in Figure 4-7. The consolidation pressures of 500, 1000 and 2000 lb/ft^2 were converted to depth of soil and were plotted against the corresponding permeability values. A linear fit was imposed on the data points and the line of fit equation is shown in Figure 4-7. Therefore, for a given soil depth the corresponding permeability values can be interpolated or extrapolated from Figure 4-7.

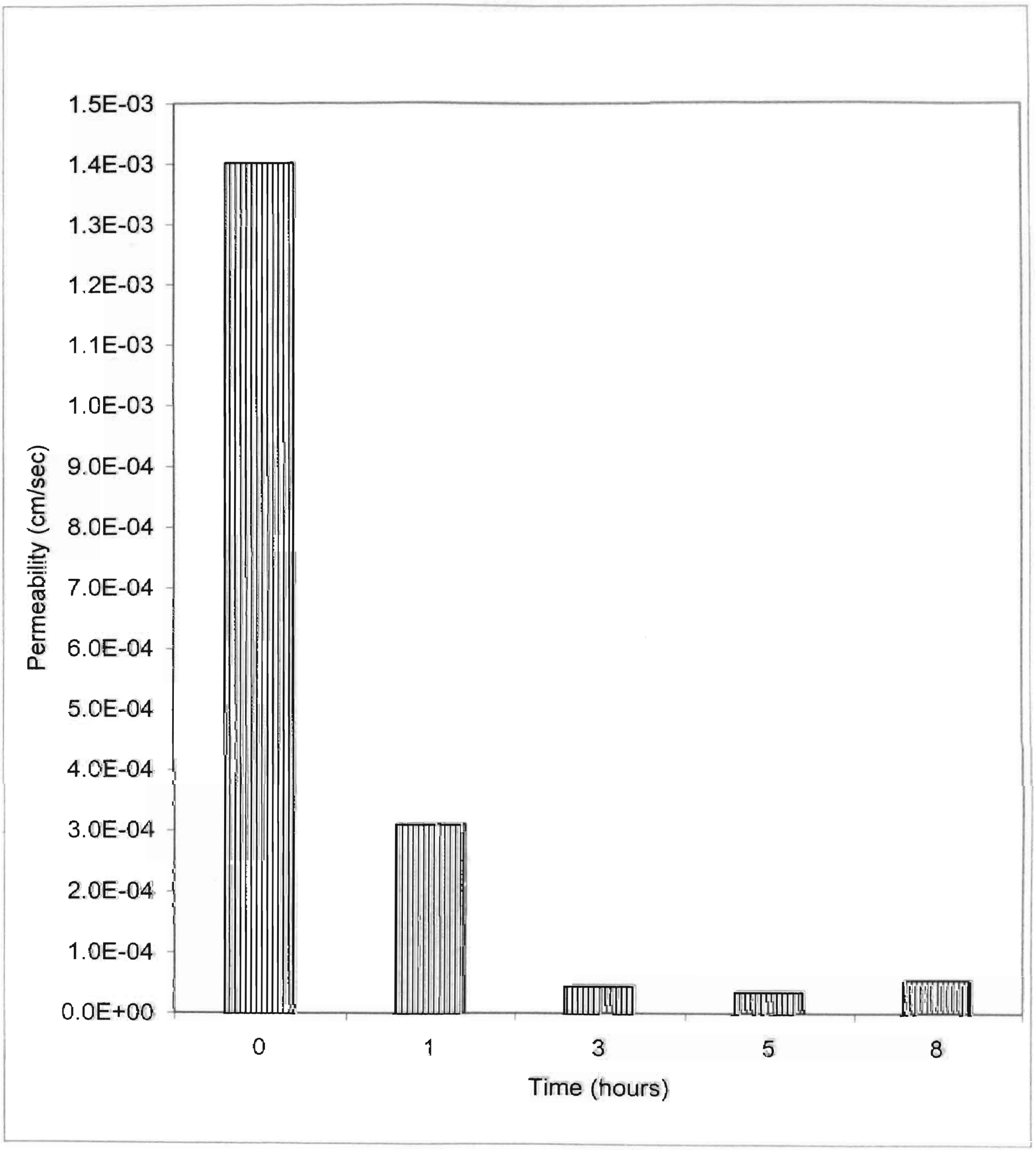

Figure 4-6 Permeability at Pre-consolidation Pressure of 2000 lb/ft^2

The relationship between consolidation pressure and permeability are shown in Table 12 and graphically presented in Figure 4-8. In Figure 4-8, logarithmic values of the consolidation pressure were plotted with permeability values. The general permeability values for various types of soil are shown in Table 13.0. The permeability of beads when compared to the general permeability values in Table 13 ranges from the medium permeability (fine gravel and sand), to low permeability (silt sand and admixtures). The application of beads in subsurface treatment depends on the subsurface permeability and the overburden pressure that will act on the bead barrier. Equation from Figure 4-8 can be used to determine the permeability of beads, if the overburden pressure is known in a given site. If the calculated permeability is greater than or equal to the subsurface permeability, then the beads can be an ideal permeable barrier.

Table 12 Average Permeability and Consolidation Pressure

Consolidation pressure (PSF)	Consolidation pressure as soil depth (ft)	Log (Consolidation pressure)	Average Permeability (cm/sec)
500	5	2.70	3.84E-02
1000	10	3.00	1.91E-02
2000	20	3.30	3.70E-04

Table 13 Typical Values of Permeability from Venkatramaiah, 1993.

Soil description	Coefficient of permeability (mm/sec)	Degree of permeability
Coarse gravel	greater than 1	High
Fine gravel - fine sand	1 to 1E-2	Medium
Silt sand, admixtures, loose silt	1E-2 to 1E-4	Low
Dense silt, clay silt, non homogeneous clays	1E-4 to 1 E-6	very low
homogeneous clay	less than 1 E-6	almost impervious

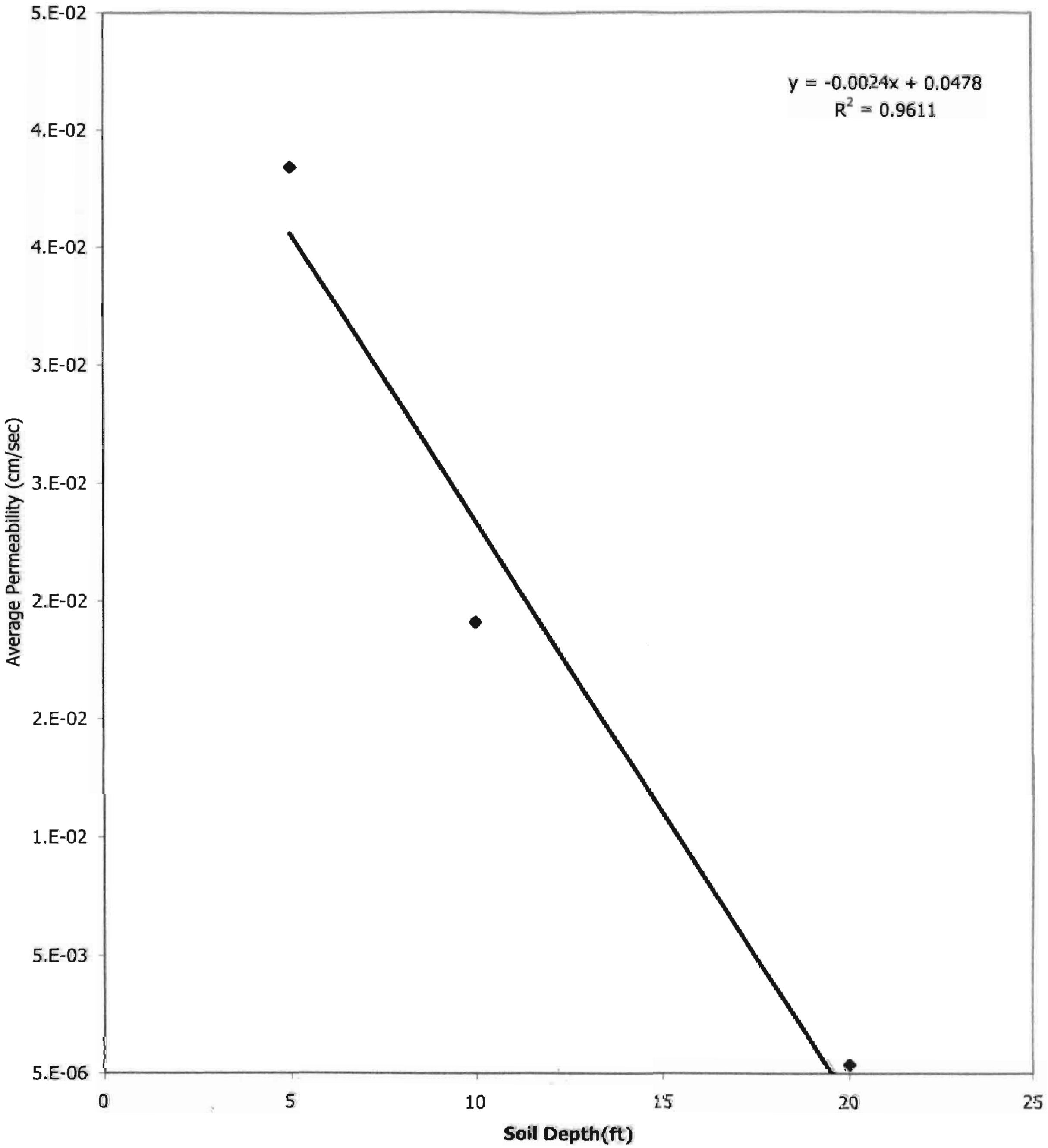

Figure 4-7 Average Permeability and Hypothetical Soil Depth

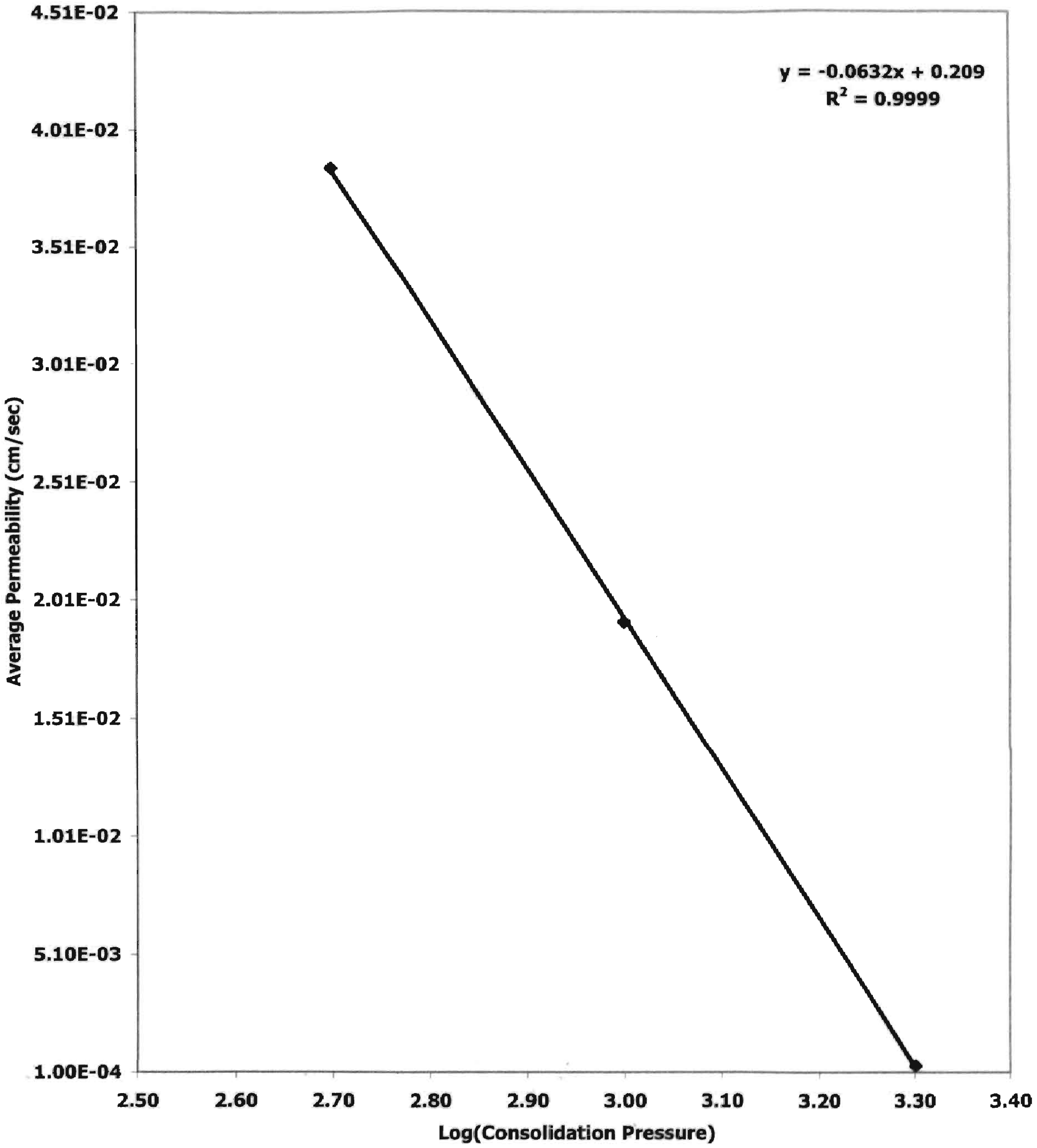

Figure 4-8 Average Permeability and Consolidation Pressure

4.2.3 Density

The density of PVA beads was calculated for 2 mm diameter beads, 4 mm diameter beads and one set of transparent beads of 4 mm diameter. The beads used during the TCE adsorption experiment had turned transparent. Therefore, those beads were tested for density to check whether the change in physical nature of beads, turning transparent, affected the density. The density results for 2 mm, 4 mm and 4 mm transparent beads are shown Table 14, Table 15 and Table 16, respectively.

The average density of 2 mm diameter beads was found to be 1.06 gm/mL. The average density of 4 mm normal beads and 4 mm transparent beads were found to be 1.07 and 1.08 gm/mL, respectively. The percentage variation in average density of the 2 mm diameter beads with respect to 4 mm diameter normal beads and 4 mm diameter transparent beads are 1 % and 2 % respectively. The percentage variation between the 4 mm diameter normal beads and 4 mm diameter transparent beads was less than 1 %. Therefore, the density of beads did not vary with respect to size or physical nature.

The densities of beads in all the three cases were found to be greater than the density of water (1 gm/mL) at 5°C. Both the normal as well as the transparent beads had similar density.

Table 14 Density of 2 mm Diameter Beads

Mass of beads (g)	Initial volume of water (mL)	Final Volume of water (mL)	Change in volume of water (mL)	Density of beads (g/mL)
5.091	30	35	5	1.02
5.101	40	44.5	4.5	1.13
5.099	50	55	5	1.02
10.201	30	39.5	9.5	1.07
10.192	40	49	9	1.13
10.112	50	60	10	1.01
15.132	30	44	14	1.08
15.154	40	54.5	14.5	1.05
15.232	50	64.5	14.5	1.05
	Average density		**1.06**	**g/mL**

Table 15 Density of 4 mm Diameter Beads

Mass of beads (g)	Initial volume of water (mL)	Final Volume of water (mL)	Change in volume of water (mL)	Density of beads (g/mL)
5.125	30	35	5	1.03
5.109	40	44.5	4.5	1.14
5.146	50	55	5	1.03
10.112	30	39.5	9.5	1.06
10.106	40	49	9	1.12
10.131	50	60	10	1.01
15.211	30	44	14	1.09
15.46	40	54.5	14.5	1.07
15.312	50	64.5	14.5	1.06
	Average density		**1.07**	**g/mL**

Table 16 Density of Transparent Beads

Mass of beads (g)	Initial volume of water (mL)	Final Volume of water (mL)	Change in volume of water (mL)	Density of beads (g/mL)
5.122	30	35	5	1.02
5.234	40	44.5	4.5	1.16
5.182	50	55	5	1.04
10.562	30	39.5	9.5	1.11
10.214	40	49	9	1.13
10.125	50	60	10	1.01
15.586	30	44	14	1.11
15.252	40	54.5	14.5	1.05
15.301	50	64.5	14.5	1.06
	Average density		**1.08**	**g/mL**

The commonly used permeable barrier materials are peat, activated carbon, and zero valent iron etc (Day, 1999). Anthracite coals specific gravity, used as a surrogate for activated carbon, was reported as 1.4 to 1.6 by Weber (1972). The zero valent iron specific gravity, reported as iron, was 7.89 by Fisher Scientific (CAS 7439-89-6).

The density of PVA beads was found to be smaller than iron and activated carbon. As the density of the beads is greater than the density of water, the beads will not float in water, instead, the beads will settle to the bottom.

4.3 Chemical Characteristics of PVA Beads

The chemical characteristics were quantified with regard to TCE and c-DCE as contaminants. The objective of this study was to determine the extent of adsorption of contaminants onto beads and the diffusion coefficient. If the PVA beads are to be used as a bio - barrier, then the loss of contaminants will be in terms of biodegradation and physical losses such as adsorption. The experiment was conducted by adding a known mass of 3.5 mm diameter beads into a 120 mL serum bottle containing distilled water and no headspace at room temperature (23±2°C).

C-DCE stock solution was added to all the serum bottles to achieve an aqueous initial c-DCE concentration of 131.72 µg/mL. The c-DCE adsorption results are shown in Table 17. Figure 4-9 shows the results of the adsorption study on PVA beads for c-DCE. From Figure 4-9, it can be seen that the concentration of c-DCE in the liquid did not show significant variation beyond 8 hours.

The samples were analyzed frequently in the first 8 hours. Seven sets of triplicate samples were analyzed during the first 8 hours. The results for the first 8 hours are shown in Figure 4-10. Between 8 hours and 48 hours, 4 set of triplicate samples were analyzed and the complete results are shown in Figure 4-9. From Figure 4-9, it can be observed that the concentration of c-DCE in liquid did not change significantly over the 48 hour period.

The mass of c-DCE in aqueous phase at the end of 8 and 48 hours were found to be 127.44 µg/mL and 123.18 µg/mL respectively. The percentage variation between the concentration of c-DCE in liquid at 8 hours and 48 hours was found to be 3.34%. Therefore it was concluded that the equilibrium condition was reached at 8 hours and hence the experiment was stopped at 48 hours. The average was calculated for the triplicate samples analyzed at 8 hours and 48 hours.

There are minor fluctuations in the concentration of c-DCE in liquid, but the average concentration in the liquid computed for triplicate samples at 48 hours varied only by 7% to the initial concentration of c-DCE in the liquid. High fluctuations in concentration of c-DCE in liquid was observed during the first 8 hour time period but, overall the adsorption on to beads was found to be minimal (7%), as the last 40 hours of data showed limited variation.

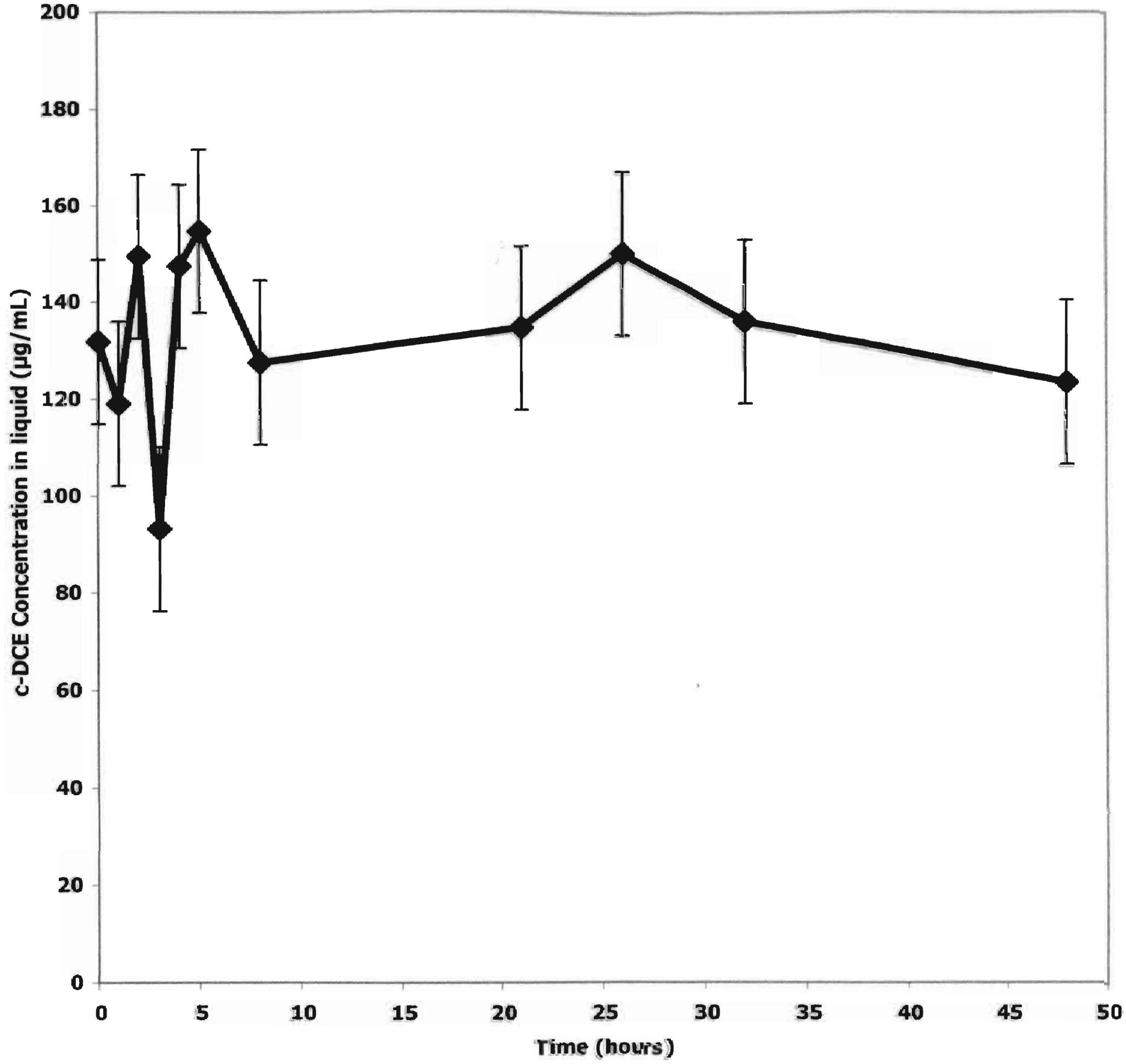

Figure 4-9 Adsorption Test on c-DCE

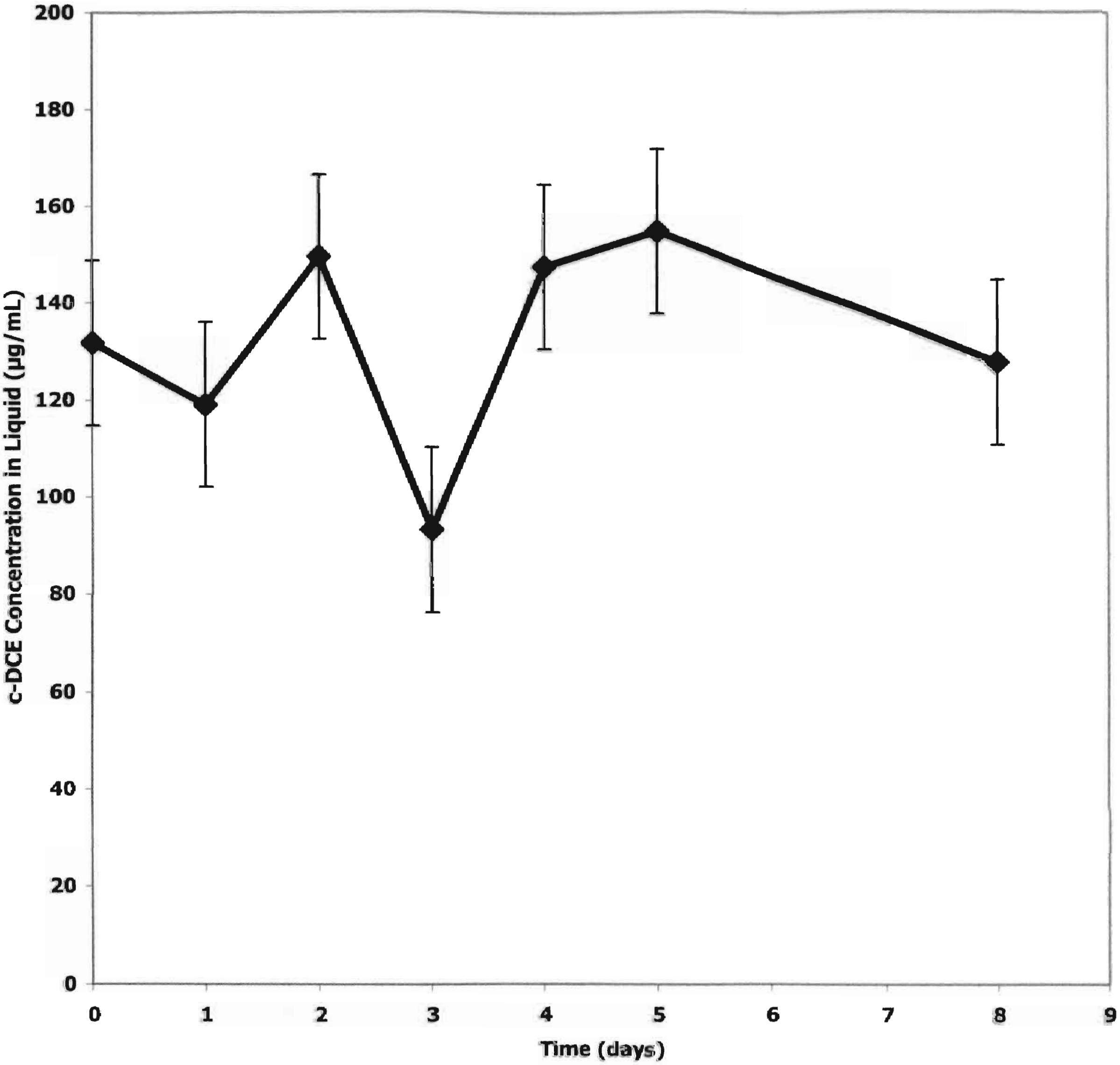

Figure 4-10 c-DCE Adsorption on PVA Beads, 8 Hours Data

The TCE adsorption study was done similarly to the c-DCE study. The samples were prepared in 120 mL serum bottle with no headspace and containing a known mass of beads. TCE stock solution was added to obtain a total liquid concentration of 112 µg/mL.

The TCE adsorption results showed high fluctuations during the first 36 hours and then remained constant beyond 36. The study was done for 150 hours. The TCE adsorption results for the 150 hours are shown in Table A.2. Figure 4-11 shows the TCE adsorption pattern during the 150 hours study and the results of first 36 hours data are shown in Figure 4-12. The first 36 hours data shows the initial fluctuations observed in the adsorption study. Similar fluctuations were also observed during the first 8 hours of c-DCE adsorption study. The results beyond 36 hours were constant and show lower values than those initial present in the system.

The concentration of TCE observed in the liquid at the start of the experiment was 84 µg/mL. The initial concentration of TCE added was 112 µg/mL, but only 84 µg/mL was detected. The calibration equation used for the analysis of TCE concentration in liquid was not a perfectly straight-line. The coefficient of variance value for the calibration equation was 0.96, which should be 1 for perfectly straight calibration line. Therefore, the measurement of 84 µg/mL at time zero instead of 112 µg/mL can be attributed to approximations involved in the fitting a linear calibration curve or it can also be due to error in the addition of TCE at time zero.

At the end of 150 hours, the average mass of TCE was found to be 67 µg. The average value at the end of 150 hours is 80 % of the initial value. The analysis of TCE concentration in liquid was done by extracting the TCE containing sample by hexane. It was assumed that extraction process with hexane will have negligible loss of TCE. Therefore, 20 % loss in TCE can be attributed to adsorption onto the surface of beads.

The initial variation in the adsorption study on c-DCE and TCE can be attributed to the phenomenon of hysteresis. According to Miyake et al. (2003), under static conditions adsorption of water vapor onto activated carbon has shown the hysteresis effect where there was continuous adsorption and desorption from the surface of activated carbon.

The initial variation can also be due to variation in the analytical instrument. Though the analytical instrument, the gas chromatograph, was used with the same conditions for all the analysis, at times it was found to vary significantly. Whenever such variations were observed, a new set of calibration standards were tested and corrections were made in the calibration equation.

The major cause for the initial variation in concentration could be due to the fact that the TCE and c-DCE in liquid were not well mixed and completely dissolved during the initial stages. With longer time duration, there is a possibility that the TCE and c-DCE could have been completely dissolved in liquid and well mixed. The 20 % adsorption of TCE and 7 % adsorption of c-DCE, respectively indicate that the beads, if used as a bio – barrier to treat TCE and c-DCE, then any observed change in TCE and c-DCE concentrations would be dominantly due to biodegradation. Langmuir and Freundlich Isotherms were plotted for the adsorption study data but a good linear fit was not obtained. Hysteresis can also be a possible cause for the variation in the mass of TCE and c-DCE in liquid observed during the initial stages of the experiment.

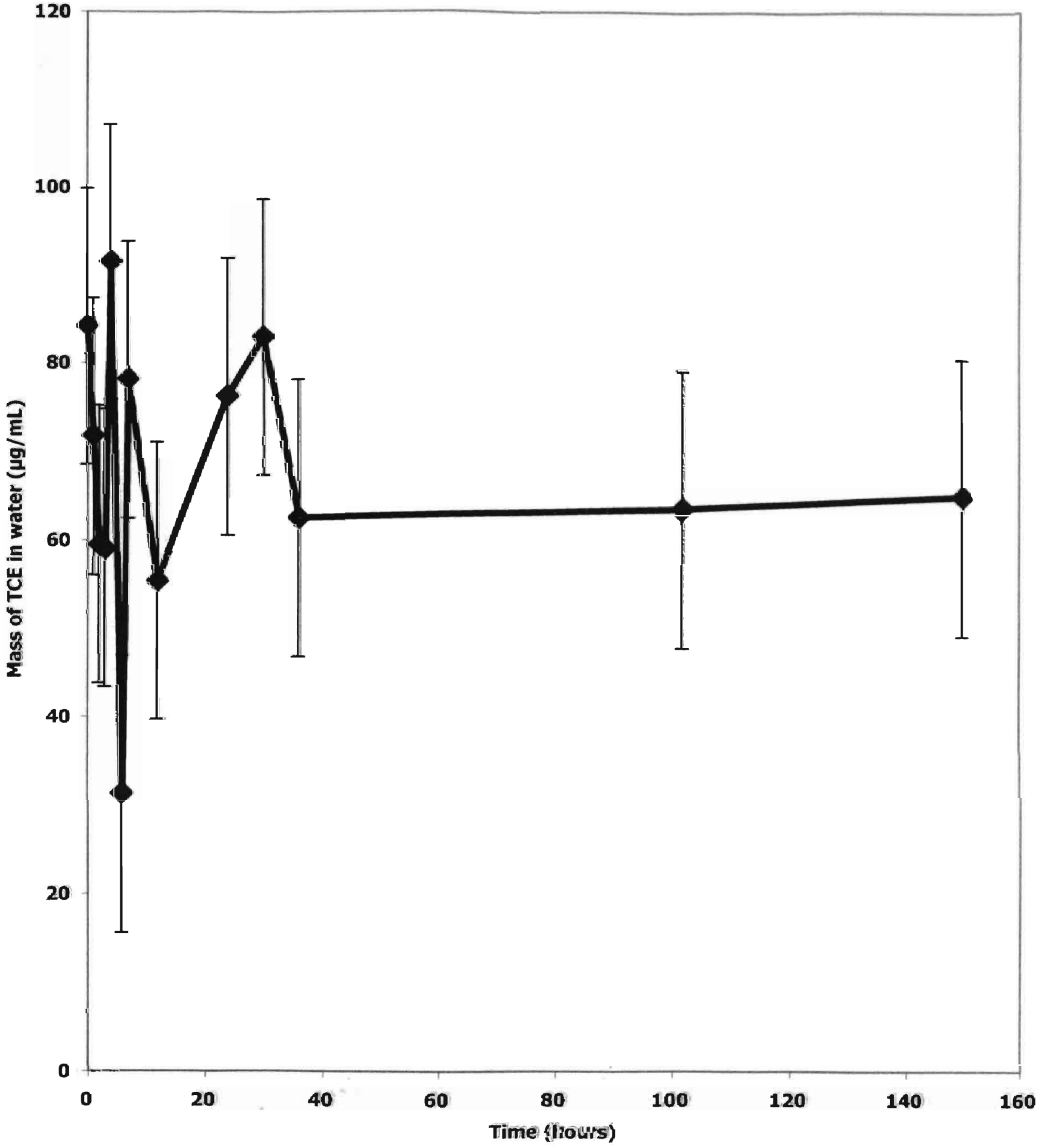

Figure 4-11 Adsorption Test on TCE

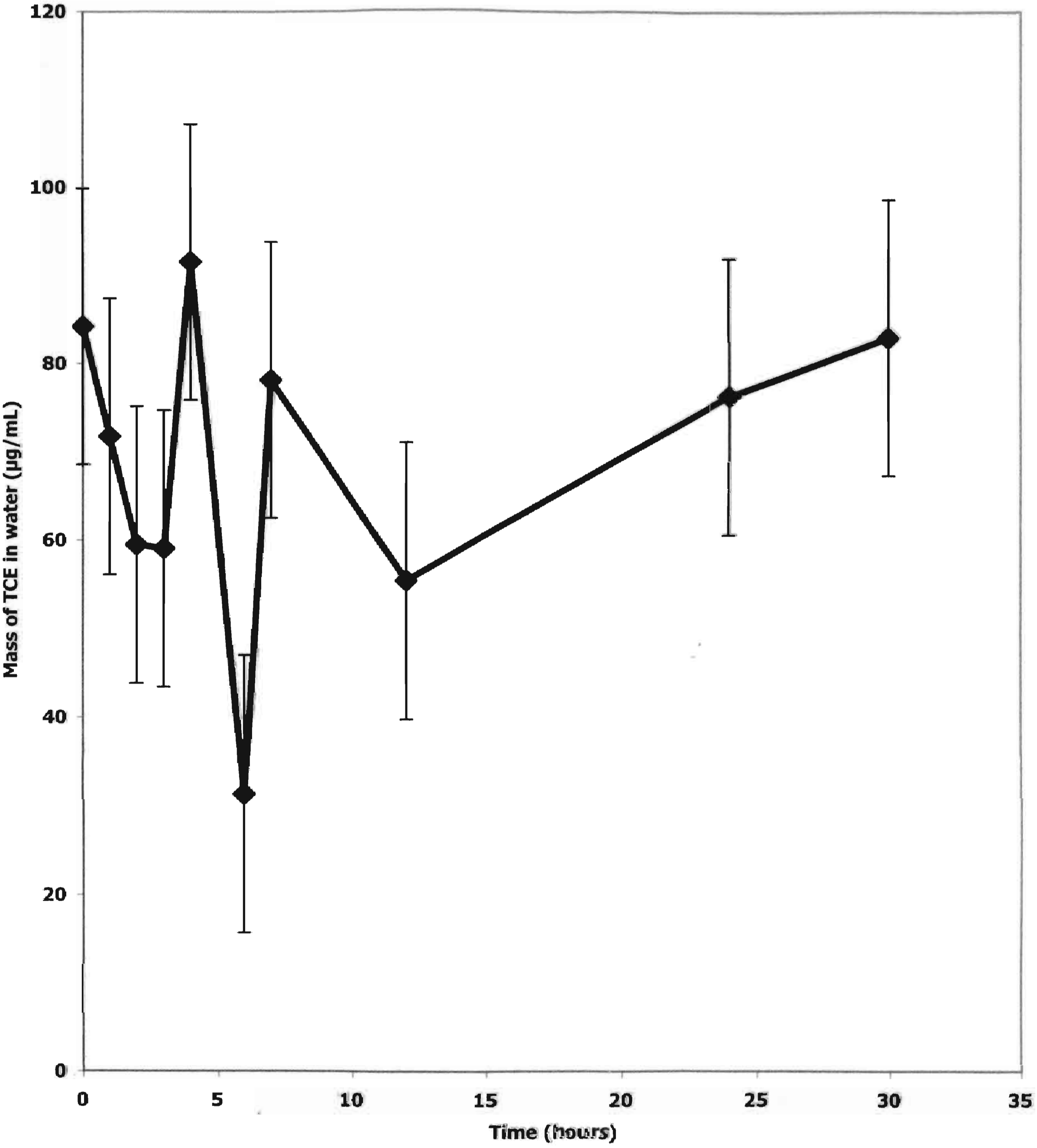

Figure 4-12 TCE Adsorption on PVA beads, 36 Hours Data

The diffusion coefficient (D) was calculated by using shrinking core model described by Chen, et al. (1993). The shrinking core model parameters for the TCE diffusion coefficient calculation are shown in Table 17. Figure 4-13 is a plot between F(X) and C*dt values from Table 17.0 and a linear fit was made for the plotted data. The slope of the linear regression curve (85.599 E-5) was used in the calculation of coefficient of diffusion.

Table 17 Shrinking core model data for TCE

Time (hours)	TCE Concentration (mg/L)	X	F(X)	C*dt
0	93.129	0.00	0.000	0
1	72.244	0.60	0.169	8.27E+01
7	66.164	0.77	0.334	4.98E+02
12	64.562	0.82	0.397	8.25E+02
102	59.91	0.95	0.688	6.43E+03
150	58.11	1.00	1.000	9.26E+03

The coefficient of diffusion for TCE in PVA beads was found to be 5.73 E-7 cm^2/s (from equation 5). Thompson (1996) reported the coefficient of diffusion of trichlorophenol (TCP) in PVA beads as 3.1E-6 cm^2/sec and Jang (1994) reported the coefficient of diffusion for Cu^{2+} in 2 % alginate beads as 1.18E-5 cm^2/s. Based on the observations by Thompson (1996) and Jang (1994), the coefficient of diffusion for TCE was found to be lesser than TCP and Cu^{2+} in beads. A coefficient of diffusion was not calculated for c-DCE as the loss of c-DCE during absorption study was only 7 %. The available date from the c-DCE absorption study was not sufficient enough to calculate the coefficient of diffusion for c-DCE. The reason for the low diffusion of TCE and c-DCE are not known.

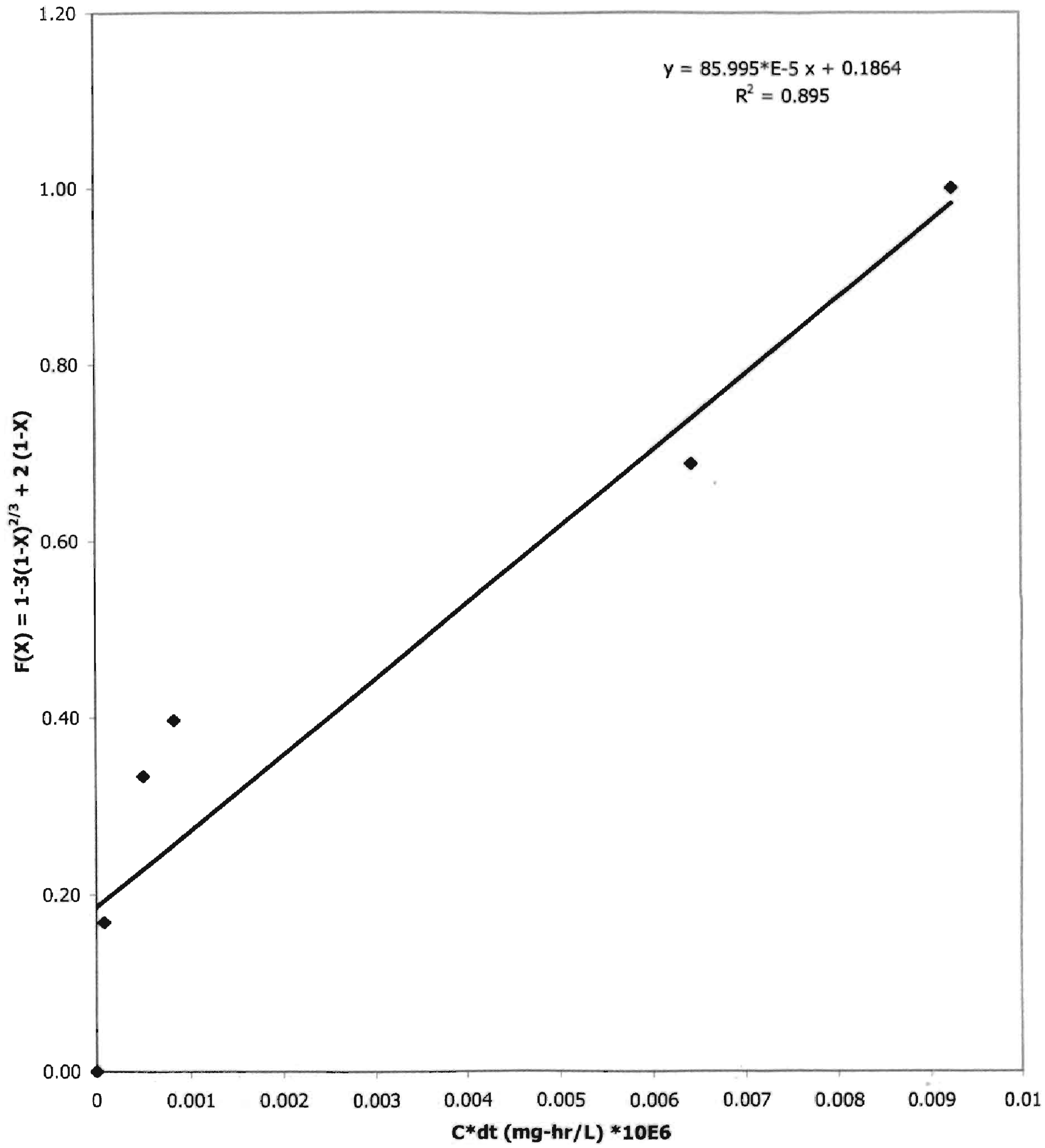

Figure 4-13 Shrinking Core Model Plot for TCE

5 Conclusion

The objective of this study was to determine the possibility of using PVA beads as a permeable barrier in groundwater remediation. Therefore, the physical properties of the PVA beads were studied to determine the ability of beads to carry overburden soil pressure and allow water to permeate through them. By conducting consolidation tests, it was observed that the beads can withstand 16,000 lb/ft^2 without collapsing, which would be 160 ft of soil depth for a soil with a density of 100 lb/ft^3. Therefore, the beads can serve as an effective barrier for an overburden pressure exerted by soil of 160 ft depth.

Permeability tests were conducted to measure the ease with which groundwater would flow when obstructed by a PVA barrier. Based on the permeability results, PVA beads had permeability in the medium - low range, which is similar to fine gravel, fine sand, silt sand mixtures and admixtures etc. The permeability of beads varied over a wide range. Importantly, the permeability of PVA beads at a consolidation pressure of 2000 lb/ft^2 was 3.72 E-4 cm/sec. The PVA beads can withstand maximum consolidation pressure of 16,000 lb/ft^2 without collapsing but the permeability at that pressure would very low. Though the permeability at a consolidation pressure of 16,000 lb/ft^2 was not determined, the permeability at consolidation pressure of 2000 lb/ft^2 was determined to be

3.72 E-4 cm/sec, which is similar to the permeability of dense silt and clay silt (Venkatramaiah, 1993). Therefore, the permeability was expected to decrease with increased consolidation loads.

The application of PVA beads as a permeable barrier depends on the permeability of the contaminated area and the overburden pressure that may act upon the bead barrier. From the obtained permeability results, the PVA beads can serve as an efficient barrier with respect to permeability for an overburden pressure of 2000 lb/ft^2 or 20 ft soil depth. Any overburden pressure greater than 20 ft depth will result in low permeability in the PVA bead barrier. If the existing site condition has as low permeability as that of beads for consolidation pressure greater than 20 ft soil depth, then the beads can serve as effective permeable barrier.

The density of various sized beads was determined and it was found that the physical properties of beads were not significantly different with respect to the size of the beads. The density of beads of 2 mm diameter and 4 mm diameter where observed to be 1.06 g/mL and 1.07 g/mL, respectively. The density of beads is greater than 1 and that could allow the beads to settle to the bottom of water instead floating in water.

Adsorption studies were conducted on the beads to test for possibility of loss of contaminants on the surface of PVA beads. Adsorption studies conducted for TCE and c-DCE showed 20% and 7% adsorption, respectively. For c-DCE adsorption, the percentage difference between the mass of c-DCE in the liquid at the start of the experiment and at the end of the experiment, after 48 hours, was only 7%. The percentage difference in mass of TCE between initial and final time was found to be 20%. Therefore, if these beads are used as permeable reactive barrier for the treatment of chlorinated solvents in groundwater, the loss of contaminants would be more likely due to biotic processes by the immobilized microorganisms, than abiotic process like adsorption.

From this study, based on the consolidation, permeability, density and adsorption results, PVA beads are found to be a good permeable barrier material for the remediation of contaminated groundwater.

6 Recommendations

Microorganisms that can degrade chlorinated solvents can be immobilized into PVA and the performance of PVA beads as a bio - barrier can be evaluated. A bio - barrier could be an effective treatment technology as there could be complete conversion of chlorinated solvents to simple non toxic chemicals. Other physical treatment process like adsorption or air stripping will convert TCE from on form to another whereas the bio - barrier technology could completely degrade and convert it to less toxic chemicals.

During this study, the boric solution that was used in the preparation of beads had pH of 3, which is considered to be too low for bacterial growth. Alternative bead making techniques can be investigated to maintain optimum pH range of 7.0. Thompson (1996) mobilized aerobic organisms to degrade TCP by the same process and was able to prove that organism can survive under such low pH condition. The growth conditions for different microorganisms can vary and therefore, the survival of organisms that can degrade TCE under such low pH conditions needs to be verified.

The present study focused on adsorption of c-DCE and TCE at only one concentration. Various concentration ranges of c-DCE and TCE in liquid can be studied to determine the effect of concentration on adsorption onto beads. The adsorption study was also done with beads of 3.5 ± .5 mm diameter. Adsorption study can be run on beads of varying sizes to determine effect of size of beads and adsorption. Adsorption and diffusion studies can be conducted on other chlorinated compounds such tetrachloroethylene, trichloromethane, trichloroethane, trans- dichloroethylene etc.

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Appendix

Appendix A

Table A. Adsorption results for c-DCE

Time	Mass of c-DCE in water (µg/mL)
0.00	131.72
1.00	118.97
2.00	149.38
3.00	93.07
4.00	147.38
5.00	154.64
8.00	127.44
21.00	134.47
26.00	149.98
32.00	135.89
48.00	123.18

Table A.2. Adsorption results for TCE

Time (hours	Average TCE mass in water (µg/mL)	Average TCE mass adsorbed on beads (µg/mL)
0.00	84.22	0.00
1.00	71.76	12.46
2.00	59.50	24.71
3.00	59.07	25.15
4.00	91.53	-7.31
6.00	31.34	52.88
7.00	78.17	6.05
12.00	55.40	28.82
24.00	76.27	7.94
30.00	82.99	1.22
36.00	62.51	21.70
102.00	63.52	20.70
150.00	64.85	19.37

VITA ②

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Master of Science

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