JOURNAL OF
ENVIRONMENTAL HYDROLOGY
The Electronic Journal of the International Association for Environmental Hydrology
On the World Wide Web at http://www.hydroweb.com
VOLUME 17 2009

LABORATORY COLUMNS: A GUIDE TO CONSTRUCTION, PACKING AND PRELIMINARY TESTS

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Due to easy operation, low cost and the useful information provided, laboratory columns packed with sediments are widely used for saturated transport studies. These include studies of hydrochemistry, environmental impact, contamination, chemical reaction kinetics, and preservation of water resources. The wide use of experimental miscible tracer displacement does not usually include descriptions of the steps followed in constructing, packing, and calibrating the columns. The fact is that the various tasks involved in column construction, if not carefully planned and executed, may lead to results that are difficult to interpret or are downright meaningless, let alone a waste of time. Based on previous experience, this paper describes the sequential steps in column construction and packing, as well as the type of tests that need to be carried out before using the column.

INTRODUCTION

Most investigations of contamination of groundwater resources, whether the source of the contaminant is natural or man-made, rely on the knowledge of the mechanisms and parameters that govern the movement of risky solutes in saturated media. A lack of information on makes it virtually impossible to suggest corrective actions, or even to define the migration of a contaminant front in time and its spatial distribution. Indeed, to make predictions, hydrogeologists need information about the system dispersive features (coefficient of dispersion, dispersivity), the relative velocity of the solute with respect to the carrier fluid (retardation factor), the existence of sorption-desorption phenomena (distribution or partition coefficient), and a clear idea of the mechanisms that drive them. They also need evidence about first-order modifying phenomena (precipitation, dissolution, ion exchange), and/or the nature of time-dependent chemical reactions (kinetic constants).

Unfortunately, just a few of the above mentioned characteristics can be identified and quantified in the field. A simpler and much less costly approach is to resort to laboratory columns, which simulate aquifer conditions at a reduced scale. Countless numbers of scientific reports clearly demonstrate the efficiency and low cost of such experiments. The results obtained are sound, comparable, and consistent with data independently verified. Despite those advantages, the literature does not contain many references related to the precautions to be taken before launching column experiments, in particular the column construction, its packing, and the initial tests to be carried out. That is precisely at the heart of this paper, which aims to save time and avoid frustrations for researchers who do not have much previous experience in handling packed columns.

COLUMN CONSTRUCTION

The basic elements of a multi-purpose laboratory column are shown in Figure 1, and the following paragraphs will make frequent references to this schematic display. It is assumed that the flow takes place from bottom to top.

The first aspect to be discussed relates to the column material. Many columns are made out of glass (regular or heat-resistant when high-temperature fluids will be used). If there is a chance that the solute can react with the glass (such as the case of fluoride or fluorosilicates), the use of plexiglass is recommended, although that may require special tools and drill bits for plexiglass cutting and drilling. For some experiments, the use of PVC could be a good alternative. If possible, the material should be transparent so as to provide an adequate control at packing time.

The column diameter has to take into account the flow that circulates and the desired packing. For example, if the column is 8 cm internal diameter packed with sediments with a mean effective porosity (θ) of 40%, and for a flow (Q) of 10 cm^3/min, the mean flow velocity (v) will be:

$$v = Q/A\ \theta = 10 / [(\pi(8/2)^2(0.4)] \cong 0.497 \text{ cm/min} \cong 2{,}614 \text{ m/year} \qquad (1)$$

Given that typical mean flow velocities in aquifers are in the range of 50-200 m/year, the above estimated flow velocity is quite high. In this case, a wise option would be to consider columns of larger diameter and/or to reduce the flow rate. Column diameter smaller than 1.5 cm is not recommended because of the boundary effects close to the column wall.

The choice of the column length depends on the need to sampling the circulating fluid at intermediate positions (Figure 1) before it reaches the outlet. It has to be noted that in a restricted

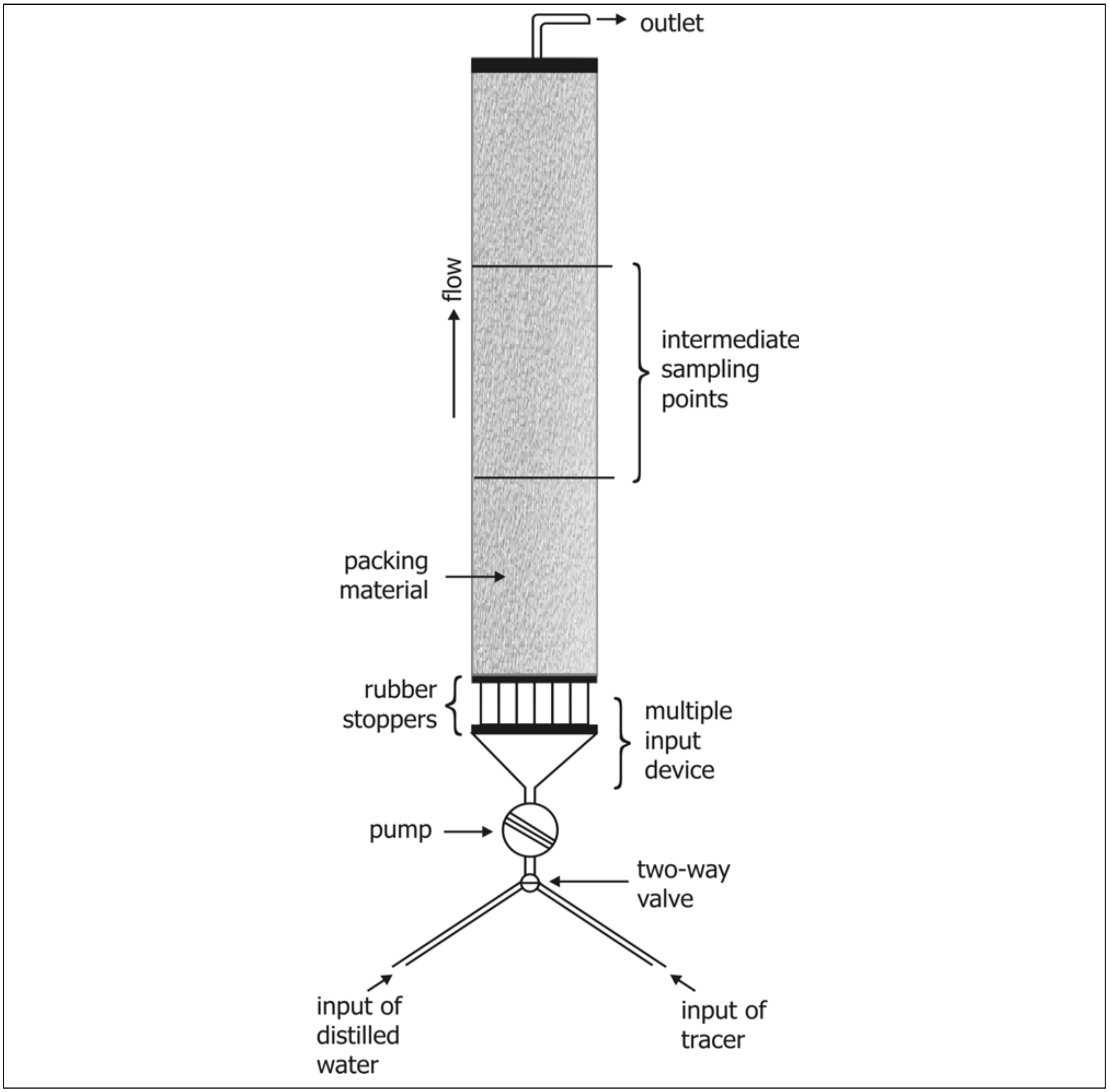

Figure 1. Typical elements of a column used in laboratory experiments.

flow domain (such as the column) any major withdrawal may alter significantly the hydrodynamic conditions. As a rule of thumb, the rate of sampling should be no more than 20% of the flow inside the column. If more than one sample along the pathway is required, the distance between contiguous sampling points should be calculated so that the sample downstream is not collected until the sampling upstream is finished.

The sampling ports (Figure 2) are T-shaped rigid plastic tubes of small diameter that lay across the column diameter. These tubes should have numerous holes and be covered by a fine weft fabric to avoid the capture of packing sediments. The two outlets of the sampling port serve a double purpose: (a) to get a sample of the circulating fluid, and (b) to measure the hydraulic head. The latter readings may be used to calculate the hydraulic conductivity between adjacent sampling points.

The multiple input device or MID (Figure 1), details of which can be seen in Figure 3, is needed to ensure the homogeneous distribution of flow at the input surface (i.e., the bottom of the

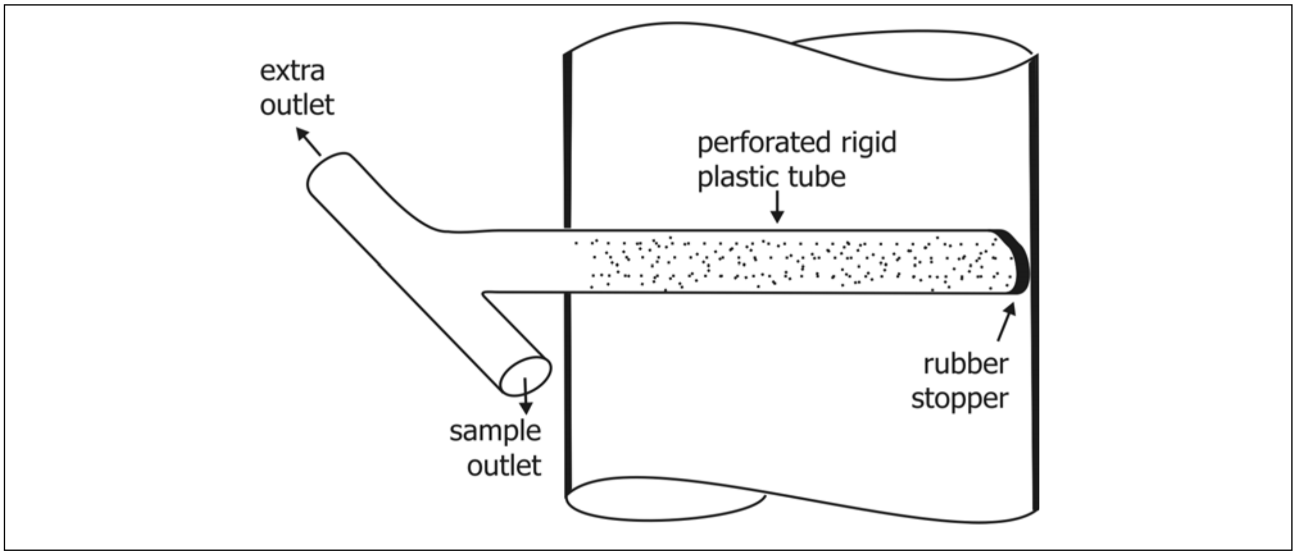

Figure 2. Sampling ports scheme.

column). Such homogeneity is required so as not to introduce components of hydrodynamic dispersion other than those legitimately exhibited by the system and, ultimately, to justify one of the primary assumptions of dispersion tests in columns such as the achieving of a “square-shaped” input wave. For that reason, the MID is connected to the bottom of the column by multiple tubes. However, the MID (placed between the container carrying the tracer solution and the bottom of the column) has certain characteristics (volume, number and diameter of tubes) that may complicate the estimation of the time that takes the tracer to arrive to the bottom of the column. Therefore, as explained below, careful MID calibration is in order.

MULTIPLE INPUT DEVICE CALIBRATION TESTS

The MID must be calibrated before putting it to use, a task that requires carrying out two types of tests.

Test of homogeneous input

Due to the particular geometry of each MID and the dispersion inside it, the tracer will select preferential paths in its upward movement, as a result of which some tubes will conduct the tracer faster than others. In order to have the tracer circulating through all the tubes and reaching the input surface (column bottom) simultaneously, the length of some tubes (or even the diameter) should be varied. The question of which tubes will be affected by this procedure is solved by the injection at a low flow rate of a dye flowing through the water-saturated MID.

Test of arrival time

Assuming that water (or any other solution not carrying the tracer) is circulating through the system and that at a given time the double-position valve is switched to allow the entry of the tracer, it will take a certain time for it to reach the input surface ($t = 0$ for the experiment). Such circulation time can simply be calculated by dividing the volume of the MID by the flow rate. In most real situations, however, the implicit assumption of piston flow may not hold, and a more refined approach is to used to calculate the arrival time (t_a). One way of doing it is using a conductivity meter and the set up shown in Figure 3. Water is circulated at a known rate Q and, at a given set time, a solution containing a strong electrolyte at high concentration (1,000 mg/l of NaCl, for example) is injected, after which well-stirred samples are taken at the outlet (base of the column) and their

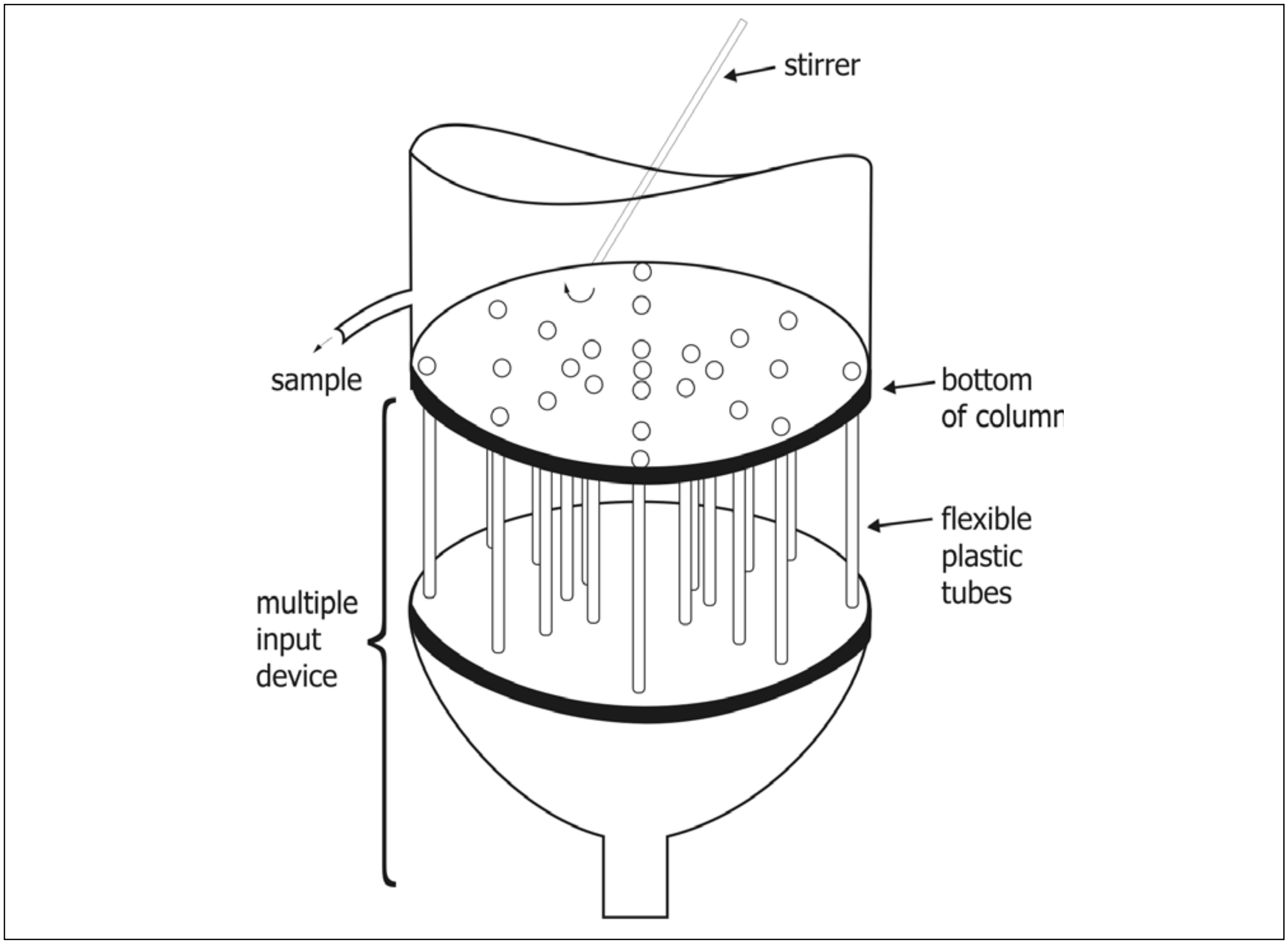

Figure 3. Elements of the multiple input device.

electrical conductivity C measured. Inasmuch as the conductivity of the input solution (C_m) and that of the background solution (C_b) are known, the relative concentration (C_r) can be calculated as follows:

$$C_r = (C - C_b) / (C_m - C_b) \tag{2}$$

If the values of C_r are the plotted against time, a typical sigmoidal curve is obtained. The test is repeated at several Q´s to cover the range of discharges to be used later in the column experiments, which will provide a set of curves of C_r versus time for a given flow rate. By reading on those curves the time value (t_a) corresponding to $C_r = 0.5$ (or 0.9 using a conservative approach), a new plot can be made of Q versus t_a to be used as the calibration curve for the MID.

COLUMN PACKING

Once the MID has been calibrated and a rubber stopper has been installed at the bottom of the column (perforated for allowing the input flow and covered by a fine weft fabric for avoiding sediments migration into the MID), the packing may start. The basic rule is that the column has to be packed in saturated conditions in order to avoid air entrapment. To do so, a known volume of water is poured into the column from its top to reach a height of about one inch inside the column. The sediment is then poured from the top (or using a tube that reaches the water level inside the column when different grain sizes may induce a preferential settling) until its level inside the column reaches that of the water. The process is repeated until the top of the column is reached. If the volume of added water has been recorded and the total volume of the column is known ("dead" spaces such as the volume of the rubber stoppers into the bottom and top sections of the column should be accounted for), the porosity can be estimated as shown below.

Measured/recorded data

Column length = 100 cm

Column diameter = 5 cm

Volume of sampling ports: $V_t = 25\ \text{cm}^3$

“Dead” volume inside the column: $V_s = 90\ \text{cm}^3$

Volume of water added when packing: $V_w = 700\ \text{cm}^3$

Porosity estimation

Total column volume: $V_c = \pi(5/2)^2 100 = 1{,}963.50\ \text{cm}^3$

Effective column volume: $V_e = V_c - V_s = 1{,}873.50\ \text{cm}^3$

Volume of water in pores: $V_m = V_w - V_t = 675\ \text{cm}^3$

Porosity: $\theta = V_m/V_e \cong 0.36$

In coming up with a value for porosity, the relationship between the dimensions of the column and the mean particle diameter can be essential to avoid errors in the estimation. Ward (1966) reported that when the ratio between the mean particle diameter and the container diameter and the ratio between the mean particle diameter and the container length were both about 0.1, an error of about 7.3% in the observed porosity was found. Air entrapment during packing can also affect the hydrodynamic characteristics of the system. Orlob and Radhakrishna (1958) concluded that a 10% increase in the air content of media voids can produce a 15% reduction in effective porosity, a 35% decrease in permeability, and about a 50% reduction in hydrodynamic dispersion. Sakaguchi et al. (2005) reported the same effect as far as the reduction of hydraulic conductivity values attributed to entrapped air. The assumption of homogeneous flow can also be incorrect for small values of the ratio between the diameter of the column and the mean particle diameter (Schwartz and Smith, 1953). Moreover, Morris and Kulp (1961) recommended the use of vibratory packers to achieve a uniform packing.

INITIAL TESTS FOR EVALUATING THE COLUMN PERFORMANCE

In order to determine the dispersive characteristics of the system, and after the column has been packed and cleaned up by circulation of excess distilled water, a dispersion test is in order. Its preparation and execution are the same as for the MID´s test of arrival time, with the advantage that t_a for the selected Q is now known. If the experiment is carried out until $C \cong C_m$ (as measured at the outlet), the plot of C_r versus time will resemble that presented in Figure 4.

Defining:

L: column length or length between input surface and sampling port [L]

Q: flow rate through the column $[L^3/T]$

A: column cross-sectional area $[L^2]$

V: mean pore velocity [L/T]

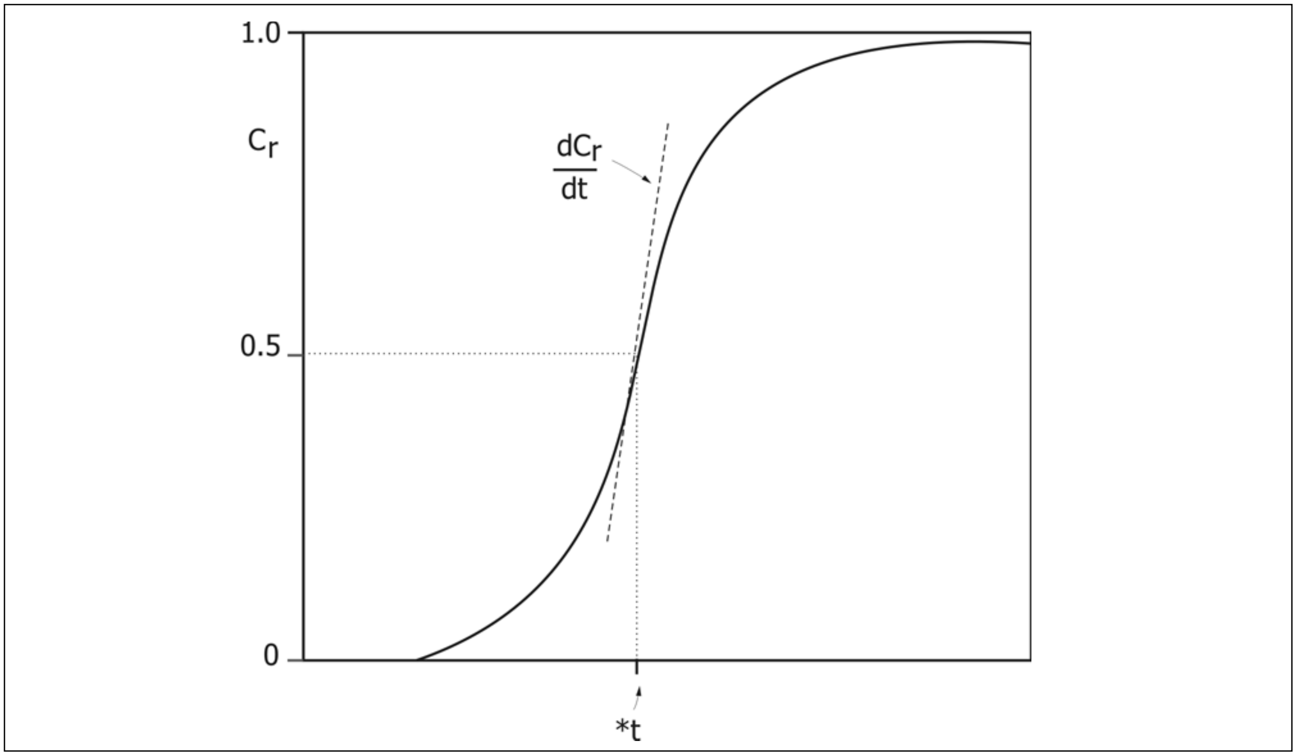

Figure 4. Typical breakthrough curve from continuous-source dispersion tests.

θ: effective porosity [L^3/L^3]

t*: x-ordinate corresponding to C_r = 0.5 [T]

dC_r/dt (at vt =L): slope of the breakthrough curve at = 0.5 [T^{-1}]

D_l: longitudinal dispersion coefficient [L^2/T]

α_l: longitudinal dispersivity [L]

Notice that L, Q, A, t*, and dC_r/dt are known, and that the other parameters can be calculated as follows (Neuman, 1984):

$v = L/t^*$

$\theta = Q/vA$

PV (pore volume) = θAL

$D_l = [v\sqrt{v}/2(dC_r/dt)]^2 (\pi L)^{-1}$

$\alpha_l = D_l/v$

It is a well-known fact that the value of the longitudinal dispersivity depends on the scale taken for its determination. In this regard, and in those cases where solute adsorption does not play a major role, α_l should be one or two orders of magnitude larger than the mean diameter (d_{50}) of the particles in the column.

REMARKS ON MISCELLANEOUS ISSUES

In adsorption-desorption studies, it is convenient to count on previous information about the partition (or distribution) coefficient K_d (USEPA, 1999) before proceeding with the column experiments. Such information may come from batch tests, in which case K_d takes on maximum values because of the nature of those experiments (i.e., the solution in contact with the sediments is not repeated). Those K_d values can be used to estimate the maximum retardation factor R (Freeze and Cherry, 1979), which will help establish the sampling sequence.

At the onset of any adsorption-desorption experiment, a basic question emerges: Is there solute adsorption in the system? If so, what are the values of the characteristic parameters? In the light of numerous bibliographic references, it would appear that column experiments are the key for assessing such a question. Once the experiments have been carried out, the use of dedicated computer codes (for instance, Simunek et al., 1999) may help in the analysis and interpretation of the breakthrough curve data.

Given that the packed column is a simplified version of an aquifer that does not interact with the atmosphere (closed system), the pH measurements of input and output solutions should reflect that restriction. It poses a double problem: the preparation of the input solutions, and the measurement of the pH of output solutions. Where the system is not open to the atmosphere, the partial pressure of CO_2 is neither constant nor unlimited. For preparing the input solutions, the following precautions should be taken (Simpson, 1986): heat up an excess volume of water until the temperature is between 80-90 °C (below the boiling point for water), which will eliminate the gases dissolved at 25 °C. Then, the tracer mass is added and the container is sealed tightly. However, be aware that:

(1) The density of water depends on its temperature. Therefore, some extra water should be added such that, in cooling down to laboratory conditions, the final volume is that required for achieving the desired tracer concentration.

(2) The container where the heated water and the chemicals are combined should be made of flexible material. Otherwise, it may collapse when the solution cools down. An effective way of overriding such negative pressures is by injecting helium (or any other inert gas) to preserve atmospheric pressure inside the container.

With respect to the measurement of pH representative of closed conditions, the objective is to avoid having the sample exposed to the atmosphere (equilibrium with atmospheric CO_2 is rapidly achieved). This may be done using a syringe of adequate volume and diameter. The sample is taken by suction, the syringe piston is taken off, and the pH-measuring electrode is introduced, sealing immediately with paraffin wax the top of the syringe, a procedure that minimizes the exposure time of the sample with the atmosphere.

In those cases where a fixed pH of input solutions is required, buffer solutions may be used, although it is convenient to estimate the potential effect of the buffer on the chemical characteristics of the system. Instead of buffering solutions, the input pH can be adjusted by adding strong acids or bases. Nevertheless, it has to be recognized that the equilibrium pH of the system is that reached inside the column, and not that of the input solution. For that reason, and before launching the column experiment, it is recommended to circulate pH-adjusted distilled water and to measure the pH at the column outlet. When the pH readings stabilize, the experiment may start.

CONCLUSIONS

It has been long acknowledged that column experiments may unravel many features related to the transport of chemical species in saturated media. They do not replace field experiments, although they provide useful preliminary information on how the hydrogeochemical system is to be conceived. Aside from those valuable early glimpses, column experiments are not expensive, can be repeated, and changes in the hydrodynamic or chemical characteristics can easily be changed.

However, an exhaustive search has demonstrated that only a restricted number of references describe or justify the column dimensions, the packing procedure, and the implementation of tests for assessing the column performance. This paper aims at fulfilling those purposes, at least partially.

Firstly, arguments for selecting the column material and size (diameter and length) are presented. Then, the precautions to be taken at packing time are discussed, including a numerical example for calculating porosity. A particular mention is made of the so-called multiple input device, and its role ensuring the homogeneous input of the tracer solution (i.e., the "square-shaped" input wave) and the estimation of the times that it takes the tracer solution to reach to the bottom of the column (that is, the true t=0 for the experiment).

An example shows the calculation of preliminary transport/dispersion parameters, such as the pore volume, the longitudinal dispersion coefficient (D_l), and the longitudinal dispersivity (α_l).

Some tips are given for the preparation and storage of the tracer solutions, and a way of measuring the pH at the outlet in order to avoid undesirable changes due to the sample exposure to atmospheric conditions.

ACKNOWLEDGMENTS

The authors would like to thank the comments and suggestions of their peers, which greatly improved the quality of the methodological issues presented. The financial support from the Universidad Nacional del Centro de la Provincia de Buenos Aires (UNCPBA) and from the Comisión de Investigaciones Científicas de la Provincia de Buenos Aires (CICPBA) has also been crucial for carrying out the projects where column experiments were carried out.

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