Strategies for Solving Potential Problems Associated with Laboratory Diffusion and Batch Experiments—Part 2: Future Improvements

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ABSTRACT

Part 1 of this study involved a brief overview of the methods used for laboratory diffusion and batch experiments. The advantages, disadvantages, limitations, and/or potential problems associated with individual tests were compared and summarized. Major conclusions drawn from part 1 of this study were as follows: 1) It is preferable to simultaneously determine the effective diffusion coefficient and the rock capacity factor directly from a diffusion test; 2) Improved laboratory through- and in-diffusion tests, specifically the decreasing inlet concentration-increasing outlet concentration through-diffusion test, decreasing inlet concentration-constant outlet concentration through-diffusion test, and decreasing inlet concentration-no flux downstream boundary in-diffusion test, have advantages over the traditional through- and in-diffusion test methods. To provide a theoretical basis for effectively organizing laboratory diffusion tests and to interpret the test results appropriately, further improvements to the individual methods were performed as described in this paper. The major improvements included in this study were as follows: 1) derivation of new theoretical solutions to the decreasing inlet concentration-constant outlet concentration test, and the decreasing inlet concentration-no flux downstream boundary in-diffusion test; and 2) sensitivity analyses for all three improved test methods as well as for the traditional through-diffusion test for comparison. The theories and approaches provided in the present study may offer practical considerations for effectively selecting an appropriate test method, designing optimum test conditions, and determining the best method of data sampling. We also provide practical references for reviewing the results obtained from a laboratory diffusion test from the viewpoint of regulation.

INTRODUCTION

Laboratory diffusion and batch experiments are very important test methods in both scientific and applied fields, including geological disposal of radioactive nuclear waste. Although many kinds of test method are currently available, different methods have different advantages and disadvantages. Paper 1 of this study presented a brief overview of laboratory diffusion and batch experiments. Major conclusions drawn from part 1 of this study were as follows: 1) it is preferable to simultaneously determine the effective diffusion coefficient and the rock capacity factor directly from a diffusion test; 2) improved laboratory through- and in-diffusion tests, specifically the decreasing inlet concentration-increasing outlet concentration through-diffusion test (DC-IC), decreasing inlet concentration-constant outlet concentration through-diffusion test (DC-CC), and decreasing inlet/source concentration-no flux downstream boundary in-diffusion test (DC-NF), have advantages over the traditional constant inlet concentration-constant outlet

concentration through-diffusion test (CC-CC) and constant source concentration in-diffusion test. These improved test methods have great potential for future applications. To provide a theoretical basis for effectively organizing laboratory diffusion tests and for appropriate interpretation of the test results, further theoretical studies on the 3 improved test methods were performed. Theoretical solutions to the individual test methods in which rigorous solutions to the DC-CC and DC-NF methods were newly derived are first discussed. The rigorous solutions were used to produce type curves and perform sensitivity analysis for each test method. These theoretical studies are of fundamental importance for the establishment of experimental design criteria and thus for obtaining high quality results from the relevant laboratory tests.

MATHEMATICAL FORMULATIONS AND SOLUTIONS

Mathematical Formulations

Schematic diagrams illustrating the conceptual models for the CC-CC, DC-IC, DC-CC, and DC-NF diffusion tests are shown in Table I. The governing equation described in this paper, as well as the initial and boundary conditions for the individual test methods are also shown in Table I, where A is the cross-sectional area of test specimen, L is the length of test specimen, C_0 is the constant source concentration to be maintained in the upstream reservoir for the CC-CC test, or the instantaneously applied initial source concentration in the upstream reservoir for DC-IC, DC-CC, and DC-NF tests, V_u and V_d are the volumes of upstream/source and downstream/measurement reservoirs, respectively, and C_u and C_d are the concentrations in the upstream and downstream reservoirs, respectively. The remaining parameters are described in paper 1.

To facilitate derivation of theoretical solutions, dimensionless variables were introduced and defined as follows:

$$c = \frac{C}{C_0} \tag{Eq. 1}$$

$$\xi = \frac{x}{L} \tag{Eq. 2}$$

$$\tau = \frac{D_e \cdot t}{\alpha \cdot L^2} \tag{Eq. 3}$$

$$\beta_u = \frac{V_u}{\alpha \cdot A \cdot L} \tag{Eq. 4}$$

$$\beta_d = \frac{V_d}{\alpha \cdot A \cdot L} \tag{Eq. 5}$$

The dimensionless governing equation, along with the dimensionless initial and boundary conditions for the individual tests can be derived as summarized in Table I.

Rigorous Solutions

Rigorous solutions to the individual tests can be obtained by the Laplace transformation method by solving the dimensionless governing equation together with the dimensionless initial and boundary conditions for the individual tests as follows:

CC-CC [1]

$$c(\xi,\tau) = 1 - \xi - 2 \cdot \sum_{n=1}^{\infty} \frac{\exp(-\phi_n^2 \cdot \tau) \cdot \sin(\phi_n \cdot \xi)}{\phi_n} \tag{Eq. 6}$$

$$\phi_n = n \cdot \pi \tag{Eq. 7}$$

Table I. Mathematical Model

Test method	CC-CC	DC-IC	DC-CC	DC-NF
Schematic of mathematical model	Upstream reservoir $C_u(t)=C_0$ — Specimen D_e, f, A — Down-stream reservoir $C_d(t)=0$; $0 \dots L$; x	Upstream reservoir V_u — Specimen D_e, f, A — Down-stream reservoir V_d; $0 \dots L$; x	Upstream reservoir V_u — Specimen D_e, f, A — Down-stream reservoir $C_d(t)=0$; $0 \dots L$; x	Upstream reservoir V_u — Specimen D_e, f, A — No flux boundary; $0 \dots L$; x
Governing equation	$\dfrac{\partial C}{\partial t} = \dfrac{D_e}{\alpha} \cdot \dfrac{\partial^2 C}{\partial x^2}$ (all columns)			
Initial condition	$C(x,0)=0 \quad 0 < x \le L$ (all columns)			
Upstream boundary condition	$C_u(t) = C_0$ $C_u(0) = C_0$	$\dfrac{V_u}{D_e \cdot A} \cdot \dfrac{\partial C_u}{\partial t} + \dfrac{\partial C}{\partial x}\bigg|_{x=0} = 0$ $C_u(0) = C_0$	$\dfrac{V_u}{D_e \cdot A} \cdot \dfrac{\partial C_u}{\partial t} + \dfrac{\partial C}{\partial x}\bigg|_{x=0} = 0$ $C_u(0) = C_0$	$\dfrac{V_u}{D_e \cdot A} \cdot \dfrac{\partial C_u}{\partial t} + \dfrac{\partial C}{\partial x}\bigg|_{x=0} = 0$ $C_u(0) = C_0$
Downstream boundary condition	$C_d(t) = 0$	$\dfrac{V_d}{D_e \cdot A} \cdot \dfrac{\partial C_d}{\partial t} - \dfrac{\partial C}{\partial x}\bigg|_{x=L} = 0$	$C_d(t) = 0$	$\dfrac{\partial C}{\partial x}\bigg|_{x=L} = 0$
Dimensionless variable	$c = \dfrac{C}{C_0}, \; \xi = \dfrac{x}{L}, \; \tau = \dfrac{D_e \cdot t}{\alpha \cdot L^2}, \; \beta_u = \dfrac{V_u}{\alpha \cdot A \cdot L}, \; \beta_d = \dfrac{V_d}{\alpha \cdot A \cdot L}$ (all columns)			
Dimensionless governing equation	$\dfrac{\partial c}{\partial \tau} = \dfrac{\partial^2 c}{\partial \xi^2}$ (all columns)			
Dimensionless initial condition	$c(\xi,0) = 0 \quad 0 < \xi \le 1$ (all columns)			
Dimensionless upstream boundary condition	$c_u(\tau) = 1$ $c_u(0) = 1$	$\beta_u \cdot \dfrac{\partial c_u}{\partial \tau} + \dfrac{\partial c}{\partial \xi}\bigg|_{\xi=0} = 0$ $c_u(0) = 1$	$\beta_u \cdot \dfrac{\partial c_u}{\partial \tau} + \dfrac{\partial c}{\partial \xi}\bigg|_{\xi=0} = 0$ $c_u(0) = 1$	$\beta_r \cdot \dfrac{\partial c_r}{\partial \tau} + \dfrac{\partial c}{\partial \xi}\bigg|_{\xi=0} = 0$ $c_r(0) = 1$
Dimensionless downstream boundary condition	$c_d(\tau) = 0$	$\beta_d \cdot \dfrac{\partial c_d}{\partial \tau} - \dfrac{\partial c}{\partial \xi}\bigg|_{\xi=1} = 0$	$c_d(\tau) = 0$	$\dfrac{\partial c}{\partial \xi}\bigg|_{\xi=1} = 0$

DC-IC [2]

$$c(\xi,\tau)=\frac{\beta_u}{1+\beta_d+\beta_u}-2\cdot\sum_{n=1}^{\infty}\frac{\exp(-\phi_n^2\cdot\tau)\cdot\beta_u\cdot(\beta_d\cdot\beta_u\cdot\phi_n^2-1)\cdot(\beta_d^2\cdot\phi_n^2+1)\cdot\sin(\phi_n\cdot\xi)}{\beta_d\cdot\phi_n\cdot\{(\beta_d\cdot\beta_u\cdot\phi_n^2+1)\cdot(\beta_d+\beta_u)+(\beta_d^2\cdot\phi_n^2+1)\cdot(\beta_u^2\cdot\phi_n^2+1)-1\}}$$ (Eq. 8)

in which ϕ_n are the roots of the following equation:

$$\tan(\phi_n)=\frac{(\beta_d+\beta_u)\cdot\phi_n}{\beta_d\cdot\beta_u\cdot\phi_n^2-1}$$ (Eq. 9)

DC-CC [2]

$$c(\xi,\tau)=2\cdot\sum_{n=1}^{\infty}\frac{\exp(-\phi_n^2\cdot\tau)\cdot\beta_u\cdot\phi_n\cdot\{\beta_u\cdot\phi_n\cdot\cos(\phi_n\cdot\xi)-\sin(\phi_n\cdot\xi)\}}{1+\beta_u+\beta_u^2\cdot\phi_n^2}$$ (Eq. 10)

in which ϕ_n are the roots of the following equation:

$$\tan(\phi_n)=\frac{1}{\beta_u\cdot\phi_n}$$ (Eq. 11)

DC-NF [2]

$$c(\xi,\tau)=\frac{\beta_u}{\beta_u+1}+2\cdot\sum_{n=1}^{\infty}\frac{\exp(-\phi_n^2\cdot\tau)\cdot\beta_u\cdot\{\beta_u\cdot\phi_n\cdot\sin(\phi_n\cdot\xi)+\cos(\phi_n\cdot\xi)\}}{1+\beta_u+\beta_u^2\cdot\phi_n^2}$$ (Eq. 12)

in which ϕ_n are the roots of the following equation:

$$\tan(\phi_n)=-\beta_u\cdot\phi_n$$ (Eq. 13)

The concentrations in the upstream/source and downstream/measurement reservoirs for the CC-CC test are kept constant but at different levels (generally 0 for the downstream side). The CC-CC test measures the time-dependent flux at the downstream boundary, $\xi=1$ ($x=L$), or total or accumulated quantity diffused into the downstream reservoir.

The time-dependent flux, $Q(\tau)$, can be calculated with the following expression [1]:

$$Q(\tau)=\frac{A\cdot D_e\cdot C_0}{L}\cdot q(\tau)$$ (Eq. 14)

where $q(\tau)$ is the dimensionless form of the time-dependent point flux and can be calculated with the following expression:

$$q(\tau)=-\frac{\partial c}{\partial\xi}(\xi,\tau)\bigg|_{\xi=1}$$ (Eq. 15)

The total quantity, $M(\tau)$, can be calculated as the time integral of the flux through the downstream boundary and can be derived as follows [1]:

$$M(\tau)=\frac{A\cdot D_e\cdot C_0}{L}\cdot m(\tau)$$ (Eq. 16)

where $m(\tau)$ is the dimensionless form of the time integral of the point flux through the downstream boundary and can be calculated with the following expression:

$$m(\tau)=\int_0^{\tau}q(\tau')d\tau'$$ (Eq. 17)

In the other three test methods, the data to be measured from individual tests are the time-dependent variations in the concentrations in the upstream and downstream reservoirs (except for the DC-NF test), and can be calculated with the following equations:

DC-IC

Concentration in the upstream reservoir:

$$C_u(\tau)=C_0\cdot c_u(\tau)=C_0\cdot c(0,\tau)$$ (Eq. 18)

Concentration in the downstream reservoir:

$$C_d(\tau)=C_0\cdot c_d(\tau)=C_0\cdot c(1,\tau)$$ (Eq. 19)

Table II. Dimensioned Parameter Range

Parameter	Minimum value	Maximum value
Effective diffusion coefficient, D_e (m^2/s)	1E-14	1E-10
Rock capacity factor, α	1E-03	1E+00
Cross-sectional area of specimen, A (m^2)	1E-03	1E-02
Length of specimen, L (m)	1E-02	1E-01
Volume of reservoir, V_u, V_d (m^3)	1E-05	1E-03
Time, t (s)	1E+02	1E+07

Table III. Dimensionless Parameter Range

Parameter	Minimum value	Maximum value
Dimensionless time, τ	1E-10	1E+04
Dimensionless volume of reservoir, β_u, β_d	1E-02	1E+05
Ratio of reservoir volume, γ	1E-02	1E+02

Table IV. Sensitivity Coefficient

Test method	CC-CC	DC-IC (DC-CC, DC-NF)
D_e	$\dfrac{\partial q}{\partial (\ln D_e)} = \tau \cdot \dfrac{\partial}{\partial \tau}(q(\tau))$	$\dfrac{\partial c}{\partial (\ln D_e)} = \tau \cdot \dfrac{\partial}{\partial \tau}(c(\tau))$
α	$\dfrac{\partial q}{\partial (\ln \alpha)} = -\tau \cdot \dfrac{\partial}{\partial \tau}(q(\tau))$	$\dfrac{\partial c}{\partial (\ln \alpha)} = -\left\{ \tau \cdot \dfrac{\partial}{\partial \tau}(c(\tau)) + \beta_u \cdot \dfrac{\partial}{\partial \beta_u}(c(\tau)) + \beta_d \cdot \dfrac{\partial}{\partial \beta_d}(c(\tau)) \right\}$

Differential concentration between the upstream and downstream reservoirs:

$$C_{diff}(\tau) = C_0 \cdot c_{diff}(\tau) = C_0 \cdot \{c(0,\tau) - c(1,\tau)\} \tag{Eq. 20}$$

DC-CC
Concentration in the upstream reservoir:

$$C_u(\tau) = C_0 \cdot c_u(\tau) = C_0 \cdot c(0,\tau) \tag{Eq. 21}$$

DC-NF
Concentration in the upstream reservoir:

$$C_u(\tau) = C_0 \cdot c_u(\tau) = C_0 \cdot c(0,\tau) \tag{Eq. 22}$$

Note that $c_u(\tau)$ and $c_d(\tau)$ can be calculated by substituting 0 and 1 for ξ in the relevant solutions to the individual diffusion tests.

PARAMETRIC STUDY
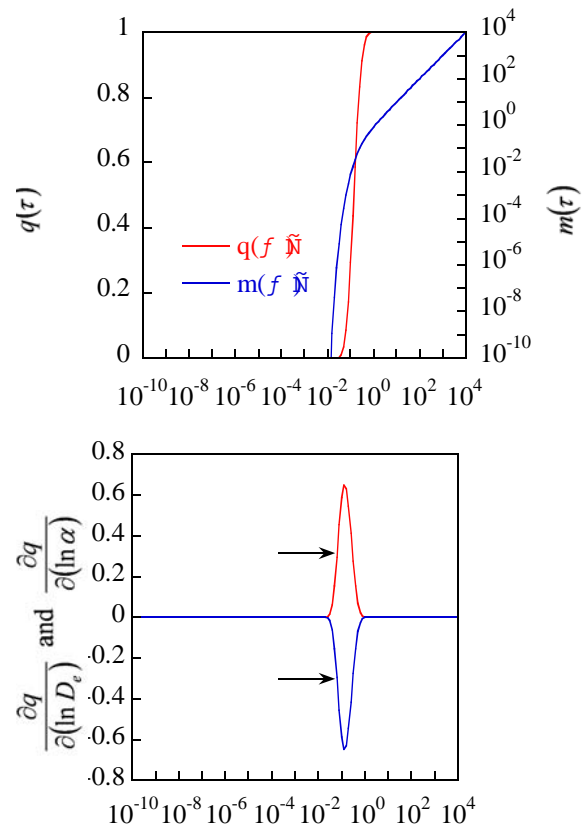
Theoretical Simulation

To examine the features of individual test methods, and to investigate the effects of system design on individual tests, a systematic parametric study was performed. The ranges for the dimensioned and dimensionless parameters were determined with reference to the test conditions and results reported in a number of previous articles and are tabulated in Tables II and III, respectively [4–7]. The sensitivities of the dimensionless measured data from relevant test methods to the logarithms of the effective diffusion coefficient, D_e, and the rock capacity factor, α, are defined by the equations given in Table IV. The type curves for the dimensionless form of the time-dependent point flux at the downstream boundary and the dimensionless form of the total diffused quantity into the downstream reservoir *vs.* time for the CC-CC test are shown in Fig. 1 a) and the sensitivity coefficients for the effective diffusion coefficient and rock

capacity factor in CC-CC test are illustrated in Fig. 1 b). The type curves for the dimensionless differential concentration, dimensionless upstream concentration, and dimensionless downstream concentration, and the sensitivity coefficients for the effective diffusion coefficient and rock capacity factor in the DC-IC test are shown in Figs. 2–4, in which γ is defined as the ratio of the volumes of the downstream r and upstream reservoirs. In addition, $\gamma = 0$ and ∞ represent the results for DC-NF and DC-CC, respectively.

Discussions

Rigorous solutions to the individual tests can be used to simulate the relevant responses from individual tests (Fig. 1 a, Figs. 2–4 upper rows). When the tracer diffuses through and reaches the downstream boundary after a certain period in the CC-CC test (Fig. 1 a), the flux at the downstream boundary increases gradually until it reaches the maximum value that corresponds to the steady state of diffusion. The accumulated quantity of tracer diffused into the measurement cell increases gradually in the transient stage and then linearly in the steady stage. The time taken for a tracer to diffuse through a test specimen depends on the effective diffusion coefficient, the rock capacity factor, and the length of the test specimen; the lower the effective diffusion coefficient and/or the larger the rock capacity factor and/or the longer the test specimen, the longer the time required for the tracer to diffuse through the test specimen. As the effective diffusion coefficient and the rock capacity

Fig. 1. (a) Type curves for dimensionless point flux, $q(\tau)$, and dimensionless total quantity, $m(\tau)$, in CC-CC. (b) Sensitivity coefficients for effective diffusion coefficient and rock capacity factor.

factor have intrinsic values for a given tracer and specimen, the only strategy to reduce the time required for testing a specimen using the CC-CC test is to use specimens that are short in length. This is why disk-shaped thin cylindrical specimens are generally used for the CC-CC test [4, 6]. However, the results obtained with thin specimens may not be representative of the properties of the rock mass *in situ*. Therefore, obtaining representative results and reducing the test time to within an acceptable duration for the CC-CC test have become important criteria for experimental design. Under simulation conditions, the sensitivity coefficients for the effective diffusion coefficient and the rock capacity factor illustrated their peak values around the dimensionless time of 10^{-1} when diffusion within the test specimen reached steady state in the CC-CC test (Fig. 1b). Therefore, the most effective data for simultaneously back-calculating the effective diffusion coefficient and the rock capacity factor seem to be the unsteady state measurements. However, the correlation between the sensitivity of the effective diffusion coefficient and the sensitivity of the rock capacity factor was 1. Thus, overestimation of the effective diffusion coefficient would cause overestimation of the rock capacity factor, and *vice versa*, by means only of unsteady state test analysis. As the flux in the steady state is independent of the rock capacity factor, it is possible to calculate the effective diffusion coefficient from the data obtained from steady-state measurements and back-calculate only the value of rock capacity factor from the unsteady state measurements.

For testing a given specimen, meaning that the dimensions and the diffusive properties of the specimen are fixed, the time required for testing the specimen can be greatly reduced by using a smaller upstream reservoir for DC-IC, DC-CC, and DC-NF tests. This is because when β_u, the ratio of the upstream/source
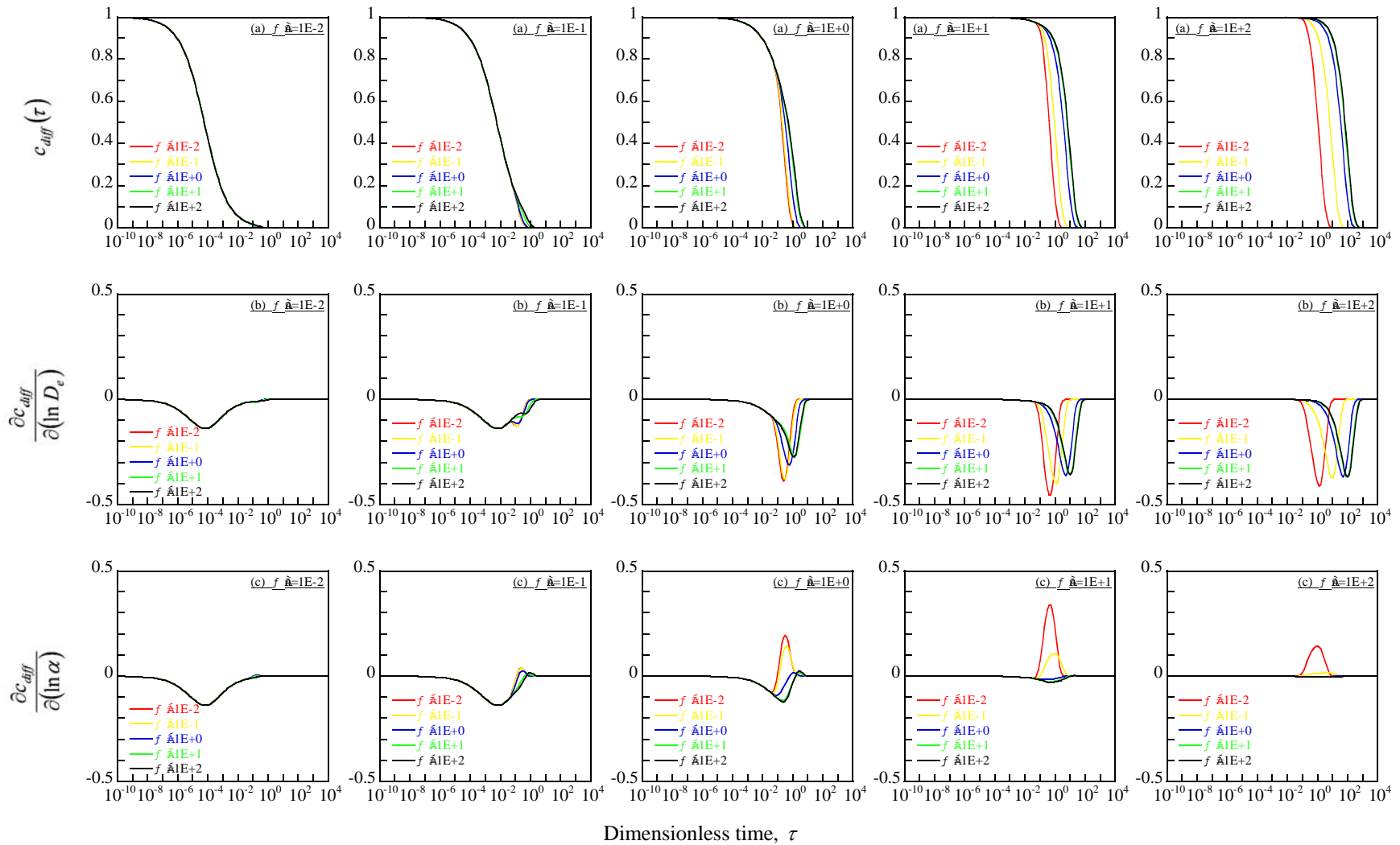
Fig. 2. (a) Type curves for normalized-differential concentrations in DC-IC. (b) Sensitivity coefficient for effective diffusion coefficient. (c) Sensitivity coefficient for rock capacity factor.
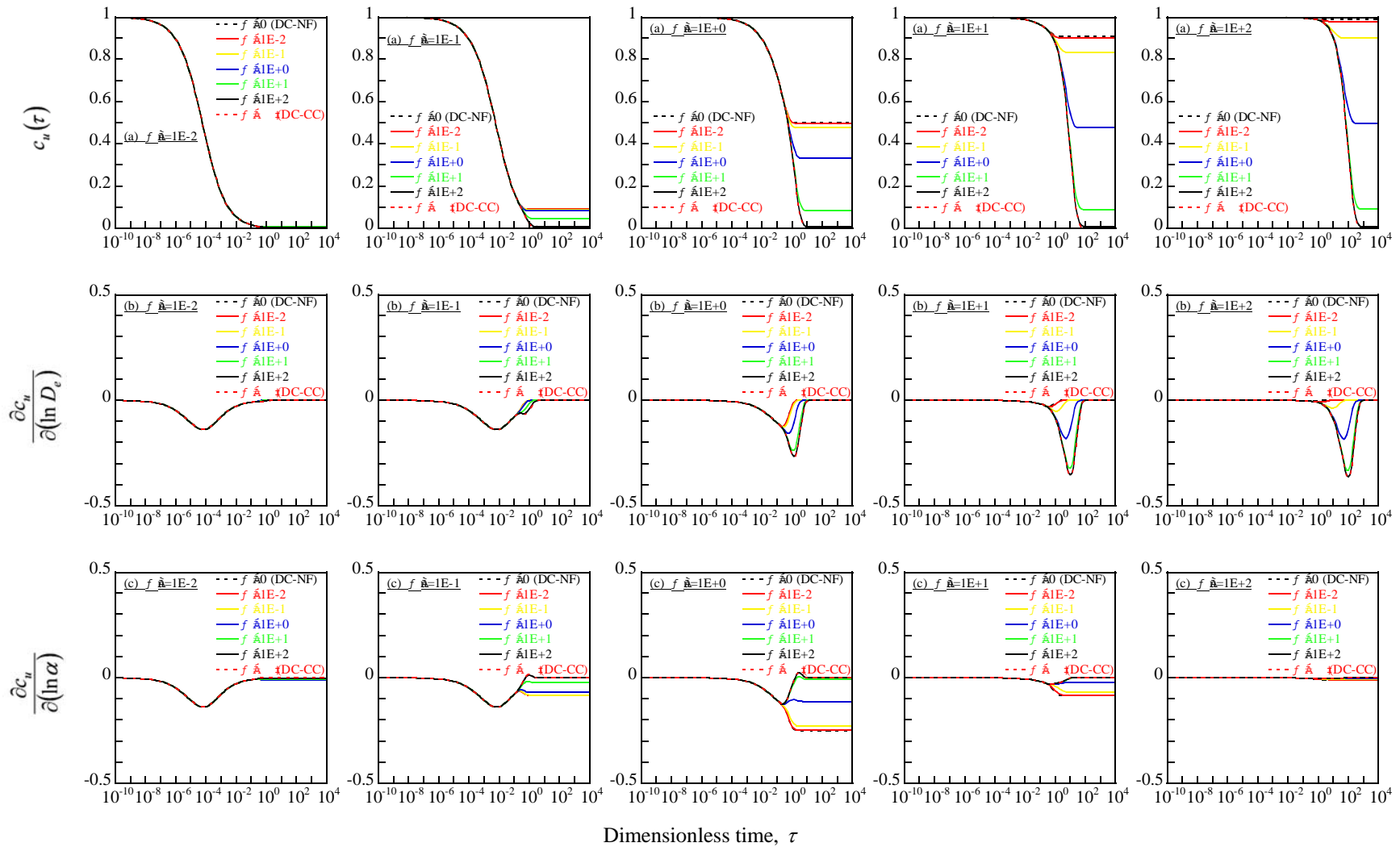
Fig. 3. (a) Type curves for normalized concentrations at upstream in DC-IC. (b) Sensitivity coefficient for effective diffusion coefficient. (c) Sensitivity coefficient for rock capacity factor.
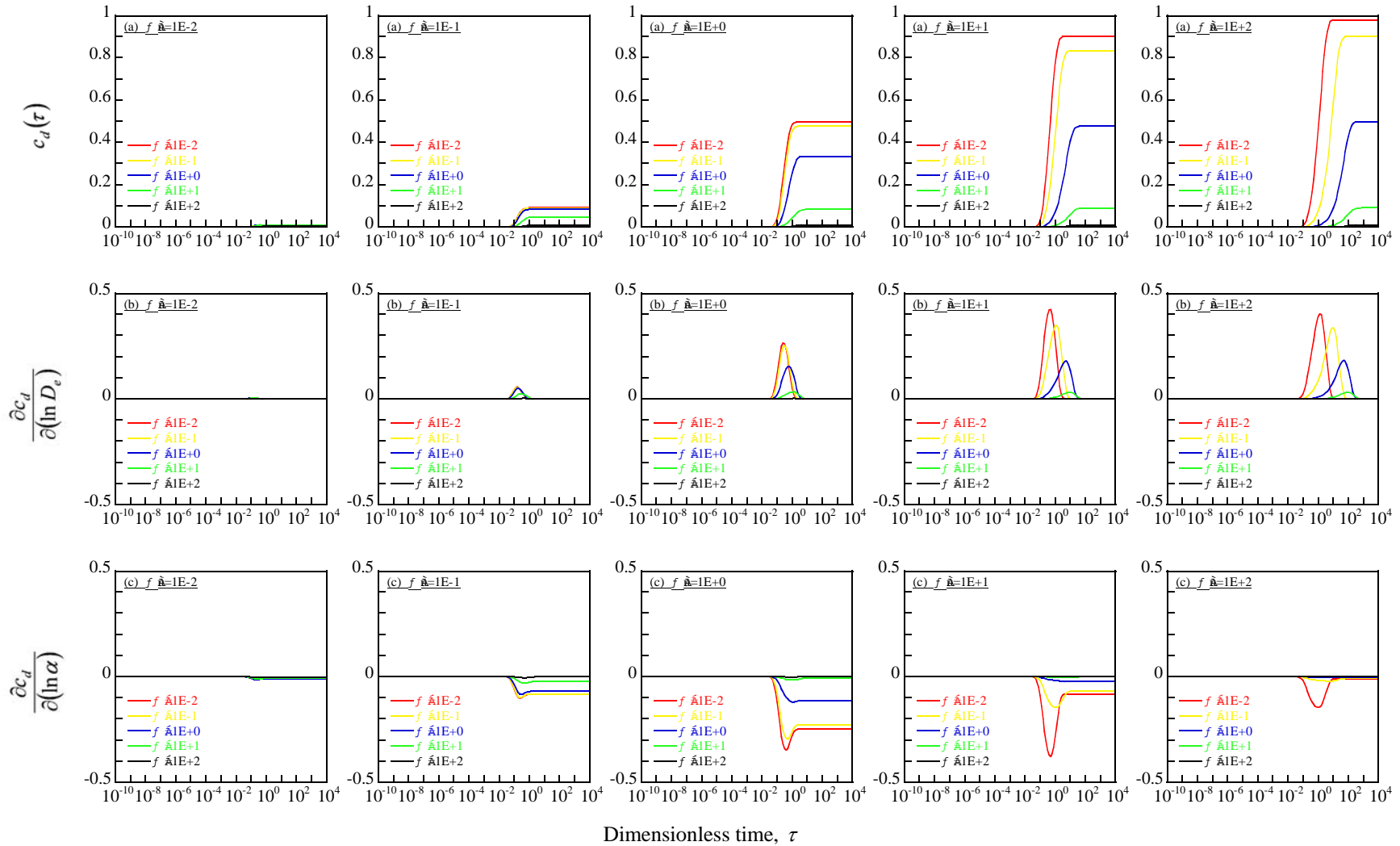
Fig. 4. (a) Type curves for normalized concentrations at downstream in DC-IC. (b) Sensitivity coefficient for effective diffusion coefficient. (c) Sensitivity coefficient for rock capacity factor.

reservoir to $\alpha \cdot A \cdot L$ (a constant for a given specimen) decreases, the dimensionless time required to reach the same value of dimensionless differential concentration, or dimensionless concentration in the upstream reservoir, or dimensionless concentration in the downstream reservoir decreases (Figs. 2–4, upper row). The volume of the downstream/measurement reservoir does not markedly affect the time required for the DC-IC and DC-CC tests, but concentration variations in the upstream reservoir are significant for the DC-CC test and thus can be detected easily (see flat lines in the upper rows in Figs. 3 and 4). In general, the sensitivities of the effective diffusion coefficient and the rock capacity factor to the concentration in the upstream reservoir and to the differential concentration between the upstream and downstream reservoirs are more sensitive than to the concentration in the downstream reservoir in DC-IC and DC-CC tests when the volume of the upstream reservoir is relatively small (Figs. 2–4, middle and lower rows, left side). When the value of β_u, corresponding to the volume of the upstream reservoir, increases and the volume of the downstream reservoir decreases, the sensitivity of the rock capacity factor to the concentration in the downstream reservoir in the DC-IC test increases (Figs. 2–4, middle and lower rows, right side). Overall, both the effective diffusion coefficient and the rock capacity factor are relatively sensitive in the case of β_u=1 for the DC-IC, DC-CC, and DC-NF tests (Fig. 3, middle column), meaning that the volume of the upstream reservoir is comparable to the total pore volume within the test specimen if sorption is not significant. This may be very difficult in practice, because design of very small-sized reservoir may not be realistic and detecting concentration variations in a very small reservoir is also impossible using current technologies. Fortunately, the sensitivity of the effective diffusion coefficient is higher in the case of β_u=10 (Figs. 2–4, second column from right). Although the sensitivity of the rock capacity factor is relatively low compared to that of the effective diffusion coefficient, this test condition is considered the best for determining the value of the effective diffusion coefficient from time-dependent variations in concentration in the upstream reservoir. As the time required for a test can be maintained within an acceptable duration under this test condition, the value of the rock capacity factor can be determined separately from the equilibrium concentration in the reservoir(s).

CONCLUDING REMARKS

Paper 1 of this study presented a brief overview of laboratory diffusion and batch experiments, with a comparison and summary of the advantages, disadvantages, limitations, and/or potential problems associated with individual test methods. The major conclusions drawn from part 1 of this study were as follows: 1) it is preferable to simultaneously determine the effective diffusion coefficient and the rock capacity factor directly from a diffusion test; 2) improved laboratory through- and in-diffusion tests, specifically the decreasing inlet concentration-increasing outlet concentration through-diffusion test, decreasing inlet concentration-constant outlet concentration through-diffusion test, and decreasing inlet concentration-no flux downstream boundary in-diffusion test, have great potential and have advantages over traditional through- and in-diffusion test methods. In this study, further theoretical studies on the individual methods were performed to provide a theoretical basis for effectively organizing laboratory diffusion tests and for appropriate interpretation of the test results. The major conclusions drawn from this study can be summarized as follows:

1) Rigorous solutions derived and illustrated for the CC-CC, DC-IC, DC-CC, and DC-NF tests can be used to simulate and study the characteristics of individual tests.

2) Rigorous solutions to the individual tests can also be used to derive the sensitivity coefficients for the effective diffusion coefficient and the rock capacity factor with respect to the relevant responses to be measured from individual tests.

3) The DC-IC, DC-CC, and DC-NF tests have the advantage over the CC-CC test in that they require only a short test period.

4) β_u values from 1 to 10 are the optimum criteria for designing DC-IC, DC-CC, and DC-NF tests. Under these criteria, the value of the effective diffusion coefficient can be sensitively back-calculated

from the test data obtained from individual tests, and the value of the rock capacity factor can be calculated from the equilibrium concentration at the end of individual tests.

The theories and approaches provided in the present study offer practical considerations for the effective selection of an appropriate test method, the design of optimum test conditions, and determination of the best data sampling method. They also provide practical references for reviewing the results obtained from laboratory diffusion tests from the viewpoint of regulation. We are currently planning a series of laboratory studies to further verify the conclusions of this theoretical study.

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