



Technical Note

Measuring air–water interfacial area for soils using the mass balance surfactant-tracer method



Juliana B. Araujo^a, Jon Mainhagu^a, Mark L. Brusseau^{a,b,*}

^a Soil, Water and Environmental Science Department, School of Earth and Environmental Sciences, University of Arizona, 429 Shantz Bldg., Tucson, AZ 85721, United States

^b Hydrology and Water Resources Department, School of Earth and Environmental Sciences, University of Arizona, 429 Shantz Bldg., Tucson, AZ 85721, United States

HIGHLIGHTS

- The mass-balance method is used to measure air–water interfacial area for two porous media.
- The interfacial areas are compared to values measured with the MD-IPTT method.
- Advantages and disadvantages of the mass-balance method are discussed.

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ABSTRACT

There are several methods for conducting interfacial partitioning tracer tests to measure air–water interfacial area in porous media. One such approach is the mass balance surfactant tracer method. An advantage of the mass-balance method compared to other tracer-based methods is that a single test can produce multiple interfacial area measurements over a wide range of water saturations. The mass-balance method has been used to date only for glass beads or treated quartz sand. The purpose of this research is to investigate the effectiveness and implementability of the mass-balance method for application to more complex porous media. The results indicate that interfacial areas measured with the mass-balance method are consistent with values obtained with the miscible-displacement method. This includes results for a soil, for which solid-phase adsorption was a significant component of total tracer retention.

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1. Introduction

There are multiple disciplines that require an understanding of fluid flow in multiphase systems. The need for considering fluid–fluid interfacial areas when characterizing and simulating fluid flow is well established (e.g., Skopp, 1985; Hassanizadeh and Gray, 1993; Gvirtzman and Roberts, 1991; Reeves and Celia, 1996; Celia et al., 1998; Kawanishi et al., 1998). There are two primary methods available to measure fluid–fluid interfacial areas for porous media systems: imaging methods (e.g., microtomography) and interfacial partitioning tracer tests (IPTT). The latter method is the focus herein.

There are several alternative approaches available to implement an interfacial partitioning tracer test to specifically measure air–water interfacial area. One approach that has been used by several

investigators is termed the mass balance surfactant tracer method, or mass-balance method for short (e.g., Schaefer et al., 2000; Anwar et al., 2000; 2001). For this method, a surfactant solution is introduced into a column packed with a selected porous medium. The test apparatus is then manipulated to allow drainage or imbibition, which generates unsaturated conditions for most of the column. The surfactant (interfacial) tracer will partition to the air–water interfaces present. Monitoring of surfactant concentrations in solution, along with measurement of tracer masses obtained from extraction of porous-medium samples collected along the column length provide a means to determine the surfactant load associated with the interfacial domain. These values are then used to calculate the interfacial areas, under assumptions of monolayer coverage.

An advantage of the mass-balance method compared to other IPTT methods is that a single test can produce multiple interfacial area measurements over a wide range of water saturations. In contrast, each test for other IPTT methods produces an interfacial-area measurement for a single water saturation. Thus, the mass-balance method can save a significant amount of experiment time. The

* Corresponding author at: Soil, Water and Environmental Science Department, School of Earth and Environmental Sciences, University of Arizona, 429 Shantz Bldg., Tucson, AZ 85721, United States.

E-mail address: Brusseau@email.arizona.edu (M.L. Brusseau).

mass-balance method has been used to date only for glass beads or for simple quartz sands that have been treated to remove associated organic matter. The purpose of this research is to investigate the effectiveness and implementability of the mass-balance method for application to more complex porous media such as natural soils.

2. Materials and methods

2.1. Materials

Sodium dodecylbenzene sulfonate (SDBS) (>95% purity, Tokyo Kasei Kogyo, Japan) was used as the partitioning tracer. The aqueous solution comprised SDBS (35 mg L⁻¹) and 0.01 M NaCl. The SDBS concentration is well below the critical micelle concentration of 414 mg L⁻¹. The interfacial tension function for SDBS was measured to determine the interfacial partition (adsorption) coefficient, K_i . A series of SDBS solutions was prepared in 0.01 M of NaCl, ranging between 15 and 105 mg L⁻¹. The surface tension was measured by a Surface Tensiometer (Fisher Scientific, model 21) using the ring method. The ring method determines the force required to detach a wire loop (i.e. ring) from the surface of a liquid (Adamson, 1982). The calculated K_i value is 2.99×10^{-3} cm for the 35 mg L⁻¹ concentration used for the tracer tests.

Two porous media were used in this study. Vinton soil (sandy, mixed thermic Typic Torrifuvent), collected locally in Tucson, AZ., and a 45/50 mesh quartz sand (Accusand). Vinton soil was sieved to remove the fraction larger than 2 mm. The sand was not treated in any manner to remove naturally occurring organic or inorganic components. Relevant properties of the porous media are presented in Table 1.

2.2. Tracer test methods

An acrylic column composed of 20 stackable rings, each 1.5 cm in height and 2.8 cm in diameter, was used for the study. The vertically placed column was packed with sand or soil, and then saturated with the SDBS solution using a recirculation system based on Anwar et al. (2000). The top of the column was open to the air. The bottom ring contained a porous plate and a hydrophilic membrane, and was connected to the solution reservoir. An initial flow rate of approximately 20 ml min⁻¹ was used to equilibrate the packed column to the solution. The flow rate was reduced gradually in small increments of 0.5 ml min⁻¹ to a final flow rate of approximately 2 ml min⁻¹, to promote drainage. The column was then maintained in recirculation mode for 7 (sand) or 14 (soil) days after drainage to promote attainment of an equilibrium distribution of SDBS. Replicate columns were used for the sand and triplicate for the soil. The data presented represent the composite of all experiments.

After the designated time, each ring was removed from the column and placed into a separate glass beaker and weighed. Each sample was then extracted using 2-d propanol following the procedure used by Schaefer et al. (2000). The samples were sealed and placed on a shaker table for 3–4 hours. Aliquots of the supernatant were analyzed for SDBS concentration. The concentration of SDBS in solution was also measured. SDBS concentrations were measured using a UV–Vis Spectrophotometer (Shimadzu model 1601) at 223 nm wavelength. After extraction, the porous-medium

samples were dried in an oven to determine dry weight and water saturation.

2.3. Data analysis

The total mass of SDBS in the system (M_T) is the sum of the mass of tracer in solution (M_w), the mass of tracer sorbed by the porous media (M_{s-w}), and the mass of tracer accumulated at the air–water interface (M_{a-w}):

$$M_T = M_w + M_{a-w} + M_{s-w} \quad (1)$$

where subscripts w , a , s denote water, air, and solid phase respectively. It is assumed that the surfactant does not partition into the non-wetting phase (air). The surface excess Γ (mol cm⁻²) is related to aqueous phase concentration (C) using the Gibbs equation (e.g., Adamson 1982):

$$\Gamma = -\frac{1}{RT} \cdot \frac{\partial \gamma}{\partial C} = K_i \cdot C \quad (2)$$

where K_i represents the interfacial partition (adsorption) coefficient, γ is the surface tension (dyn cm⁻¹), C represents the aqueous phase concentration (mol cm⁻³), and R is the gas constant (erg mol °K⁻¹). For adsorption at the air–water interface, the mass loading is related to the magnitude of the air–water interfacial area, a_{a-w} (cm²):

$$M_{a-w} = \Gamma_{a-w} \cdot a_{a-w} \quad (3)$$

The total mass of SDBS for each ring (M_T) is the mass of SDBS extracted from each individual ring. The final aqueous SDBS concentration provides, along with the water saturations, the means to determine the mass of SDBS in solution present in each ring (M_w). The mass sorbed by the solids (M_{s-w}) is obtained from the saturated (bottom) ring. If the ring is saturated, it follows that there is no air–water interface present in the system. Thus, the difference between M_T and M_w provides the mass of surfactant sorbed. The mass accumulated at the air–water interface (M_{a-w}) is determined by subtracting M_w and M_{s-w} from M_T . The Γ is obtained by using the adsorption isotherm (Eq. (2)), $\Gamma = K_i C$, where K_i is obtained from the measured surface-tension function. Eq. (3) is then used to determine a_{a-w} . The a_{a-w} values are then divided by the volume of the respective porous-medium samples to determine the volume-normalized specific air–water interfacial areas, A_{aw} (cm⁻¹).

3. Results & discussion

The results of the tests are presented in Figs. 1 and 2 for the sand and soil, respectively. The data display a fair amount of scatter, consistent with prior reports (e.g., Anwar et al., 2000; Schaefer et al., 2000). The interfacial areas are generally larger for lower water saturations as would be expected. In addition, the values for a given water saturation are larger for Vinton, which is consistent with the difference in median grain diameter between the two media (Anwar et al., 2000; Costanza-Robinson and Brusseau, 2002; Cho and Annable, 2005; Brusseau et al., 2009, 2010). These results indicate that the method produced values that are consistent with what is expected.

Table 1
Relevant physical properties of the porous media.

Medium	Sand (%)	Silt (%)	Clay (%)	Median diameter (mm)	Uniformity coefficient U^a (-)	Bulk density ρ_b (g cm ⁻³)	Porosity n (-)	K_{sat} (cm min ⁻¹)
Vinton	97	1.8	1.2	0.23	2.4	1.50	0.376	0.2
Sand	100	0	0	0.35	1.1	1.65	0.326	1.3

^a $U = (d_{60}/d_{10})$.

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