



Research papers

Use of sodium fluorescein dye to visualize the vaporization plane within porous media



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ABSTRACT

The vaporization plane in porous media separates the region of capillary flow from the dry surface layer, where the water transports only in its gas phase. Knowledge of the depth and geometry of the vaporization plane is critical for estimating water flux in the soil-atmosphere interphase, for understanding evaporating processes in general, and for prediction of locations of damaging salt crystallization, etc. However, detection of the vaporization plane is a challenging task. This paper explores the use of sodium fluorescein dye (uranine), a popular hydrological tracer, to visualize the vaporization plane in porous media. Uranine was used in the forms of solution or powder on sand, sandstone, and autoclaved aerated concrete, in both laboratory and field experiments. The property of uranine solution to change its color according to its concentration can be used to: i) visualize the vaporization plane by forming a distinctive dark-orange zone where the pore water evaporates, and ii) to distinguish the zone of vapor flow from the zone where capillary flow is present. Similarly, uranine powder, when applied onto a porous material, clearly visualizes the dry surface layer and the capillary zone, divided by the vaporization plane. This technique can also visualize a complex-shaped vaporization plane in hydrophobic materials. In comparison with other techniques, such as sensible heat balance or heat pulse methods, the use of uranine is accurate, cost-effective, and straightforward.

1. Introduction

Evaporation is a key process in the land-atmosphere water balance; additionally, the near-surface flow in porous media is an important topic for natural and engineering sciences such as agriculture, soil science, hydrology, or studies of rock weathering, building structures decay, or endolithic biology. Evaporation from coarse porous media can be conceptually divided into three stages (Idso et al., 1974; Hillel, 2004; Or et al., 2013). In the third stage, a critical suction is reached in the subsurface, and the so-called vaporization plane (VP) is formed, separating the region of the capillary zone (CZ) with its capillary flow from the dry surface layer (DSL), where water flows only in the gas phase (Shokri et al., 2009; McAllister et al., 2016). In the CZ, where saturation is above the critical suction, water flows via a network of partly saturated pores due to the pressure head gradient, and its flow is usually described by Richards (1931) equation. In the DSL, the water vapor movement is conceptually given by Fick's Law (Bitelli et al., 2008). Knowledge of the DSL thickness and the geometry of the VP are critical for estimating the water flux in the soil-atmosphere interface, and thus for understanding evaporation processes in general (Shokri

and Or, 2011; Bruthans et al., 2018).

An understanding of moisture dynamics in porous media is also crucial for the prediction of material decay for both building structures and natural outcrops (Fidriková et al., 2013; Mol and Viles, 2013). Water represents one of the main degradation factors, capable of seriously reducing the durability of materials (Falchi et al., 2015), as well as being one of the most critical factors facilitating the growth of mold and microbes that can pose health hazards to people (Viitanen et al., 2010). Detection of the VP is particularly important in studying a material's decay by salt weathering. As dissolved salts are transported by the capillary water, and precipitated within the material, they form crystals (Huinink et al., 2004), with the resulting increased pressure caused by the crystallization leading to the material damage (Rijniers et al., 2005). Therefore, the location of salt crystallization in the VP is closely related to material decay. Detection of the VP could also potentially be used in the soil science of (semi-)arid regions, where salinization of the soil is an important issue, and an understanding of salt accumulation can lead to better agriculture practices (Kurtzman et al., 2016).

The VP can be located by several direct and indirect methods (see

Abbreviations: VP, vaporization plane; DSL, dry surface layer; CZ, capillary zone; BB, Brilliant Blue; AA, autoclaved aerated

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Table 1 in Suppl. mat.). As heat balance is an important factor controlling evaporation (Gupta, 1974; Mikhailov, 1975), heat-pulse or sensible heat balance methods can be used for its determination (Trautz et al., 2014). These heat-based methods are limited by needle spacing, and they are required to be installed at precisely defined depth intervals (Xiao et al., 2012). The VP can also be found by measuring the water content. These methods are easy-to-employ and inexpensive. The electric- and magnetic-based methods are either not accurate enough (time domain reflectometry method; Topp et al., 1982) or they are influenced by the amount of dissolved solids (protimeters; Mol and Viles, 2010). Identification of the VP is usually not possible by measuring the pressure head with tensiometers, as the suction is out of measuring limits. More demanding methods include nuclear magnetic resonance imaging (Reis et al., 2003; Sněhota et al., 2010; Lehoux et al., 2016), X-ray tomography (Rad et al., 2015), acoustic emission (Grapsas and Shokri, 2014), neutron radiography (Deinert et al., 2004; Sacha et al., 2015), confocal microscopy (Xu et al., 2008), gamma ray densitometry (Shahidzadeh-Bonn et al., 2007), and electrical resistivity tomography (Daily et al., 1992; Mol and Viles, 2010).

The techniques used for detection of the VP within porous media require either special devices and/or have a low spatial resolution (for details about the methods and their limits, see Table 1 in Suppl. mat.). An easy-to-use and cost-effective method with good resolution has, to date, been lacking in hydrological science. The use of dyes is an attainable means to tackle the disadvantages of the methods mentioned above. In several evaporation studies, Brilliant Blue FCF dye tracer has been used to visualize the VP (Lehmann and Or, 2009; Shokri et al., 2009). However, this has been found unsuitable for tracing the travel time of water, because of its sorption properties (Kasteel et al., 2002). Concerning other uses of dyes, uranine (sodium fluorescein) dye has recently been used to visualize the complex pattern of hydrophobicity in the biologically-initiated rock crust on sandstones (Slavík et al., 2017), and even the VP within sandstone (Bruthans et al., 2018).

The aim of this study was to test the use of uranine dye in solution and powder forms to visualize the VP, CZ, and DSL in porous media. The following steps were taken to reach the aim, that is, to test if this new method is viable in different porous media and under various boundary conditions:

- In the first step, we tested *dissolved* uranine dye to visualize the VP, CZ, and DSL zones in sand, sandstone and autoclaved concrete as examples of common natural and building materials. Correct distinction of the zones was verified by testing the material cohesion, and by examining the presence of liquid water by a microscope. At first, tests were done under semi-steady state of VP, CZ, and DSL location, later under transient conditions (increased water supply from below or increased potential evaporation resulting in migration of the zones). To test if the technique can trace former distribution of VP, CZ, and DSL in already dried material, specimens with uranine solution were dried completely and subsequently cut and inspected. Finally, the technique was tested on sandstone samples with hydrophobic layer, i.e. in complex environment in terms of capillary flow.
- In the second step, we compared the uranine and Brilliant Blue dyes' ability to visualize the VP, CZ, and DSL in sand.
- In the third step, uranine in the form of *powder* was tested in lab and field experiments to visualize the three zones in sandstone.
- Finally, the applicability of the method using uranine solution or powder, comparison with other methods, and the need for future development of the proposed technique was discussed.

2. Materials and methods

2.1. Materials

2.1.1. Dyes

In the experiments presented herein, we have used mainly uranine dye ($C_{20}H_{10}Na_2O_5$), and, in one experiment, Brilliant Blue FCF dye (hereafter BB; both obtained from Sigma Aldrich in powder form). Uranine is a dark red powder when dry, and the color of a uranine-water solution changes (with decreasing concentration) from red to orange to yellow and finally to light green (Käss, 1998; Fig. 1 in Suppl. mat.). Uranine, unlike BB, has the ability to fluoresce (i.e., adsorb shorter wavelength light and emit at a longer wavelength) at lower concentrations. BB is always blue in color: dark blue in dry powder form and lighter blue in water solution.

Uranine, discovered in 1871, is a manufactured organic compound usually used as a dye, with numerous applications ranging from medicinal use (Can Med Assoc J, 1959; Mathew, 2014) to forensics (Budowle et al., 2000), and cosmetics. In the natural sciences, uranine is used in biochemical research as a common fluorophore in microscopy (Lakowicz, 2006), and as a fluorescent probe in cytogenetic techniques (Noga and Udomkusonsri, 2002). In the earth sciences, uranine is used as a rather conservative tracer in tracer tests and for the visualization of the preferential pathway(s) of groundwater flow for its relatively low-reactive (low-sorbing) properties in comparison with other organic dyes (Gaspar, 1987; Wilson et al., 2016), even though sorption was observed to be significant on soils (Gerke et al., 2008). For a summary of the available literature on uranine's main physical and chemical properties see Gerke et al. (2013). BB is an organic compound, which besides being a food coloring, is often used as a tracer in soil-profile scale studies for the visualization of flow pathways in soils (e.g., Kasteel et al., 2002). Of the non-fluorescent tracers, Flury and Flüehler (1995) concluded BB to be the best tracer to use because of its mobility, visibility, and low toxicity. For BB's main physical and chemical properties see Flury and Flüehler (1995).

Sorption characteristics of the dyes are important for migration of dye solutions in capillary form. There are several adsorption studies of BB (e.g., Ketelsen and Meyer-Windel, 1999; Germán-Heins and Flury, 2000; Kasteel et al., 2002; Mon et al., 2006; Morris et al., 2008) and uranine (e.g., Smart and Laidlaw, 1977; Sabatini and Austin, 1991; Kasnavia et al., 1999; Sabatini, 2000; Gerke et al., 2008; Gerke et al., 2015). However, as these studies were done for different types of soils and under different settings, comparisons of these findings and predictions of a tracer's behavior in a particular experiment is difficult. Even though there is a study comparing the two dyes in their ability to stain preferential pathways (Vlček et al., 2017), to the best of our knowledge, there has never been a study purposefully comparing sorption under neutral pH of the two dyes in the same material. Nevertheless, based on the literature reviewed, we expect BB to sorb more than uranine. The basic properties of uranine and BB are shown in Table 1.

Table 1
Selected physical and chemical properties of uranine and BB (Brilliant Blue FCF).

Property	Uranine	Brilliant Blue FCF	Reference
Chemical formula	$C_{20}H_{10}Na_2O_5$	$C_{37}H_{34}N_2Na_2O_9S_3$	–
CAS number	518-47-8	3844-45-9	–
Molar mass (g/mol)	378.28	792.85	–
Color Index	C. I. 45 350; Acid Yellow 73	C. I. 42 090; Acid Blue 9, Food Blue 2	(CI, 2001)
Solubility in water	> 600 g/l	200 g/l	(Käss, 1998; Marmion, 1991)

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