Rapid and Fully Automated Measurement of Water Vapor Sorption Isotherms: New Opportunities for Vadose Zone Research

Emmanuel Arthur,* Markus Tuller, Per Moldrup, and Lis Wollesen de Jonge

Eminent environmental challenges such as remediation of contaminated sites, the establishment and maintenance of nuclear waste repositories, or the design of surface landfill covers all require accurate quantification of the soil water characteristic (SWC) at low water contents. Furthermore, several essential but difficult-to-measure soil properties, including clay content and specific surface area, are intimately related to water vapor sorption. Until recently, it was a major challenge to measure detailed water vapor sorption isotherms accurately within a reasonable time frame. This priority communication illustrates potential applications of a new, fully automated, and rapid Vapor Sorption Analyzer (VSA) to pertinent issues in vadose zone research. Detailed vapor sorption isotherms for 25 variably textured soils were measured with the VSA within 1 to 3 d. Links between generated isotherms and pesticide volatilization, toxic organic vapor sorption kinetics, and soil water repellency are illustrated. Several methods to quantify hysteresis effects and to derive soil clay content and specific surface area from VSA-measured isotherms are presented. Besides above mentioned applications, potential relationships to percolation threshold for solute diffusion in unsaturated soil and to soil cation exchange capacity are discussed to stimulate new and much-needed vadose zone research.

Accurate characterization of the soil water characteristic (SWC) at low water potentials (−10 to −450 MPa) is crucial for understanding and modeling of numerous vadose zone processes including volatilization of volatile organic compounds (VOCs) and pesticides, water vapor transport, and hysteresis (Amali et al., 1994; Chen and Rolston, 2000; Chen et al., 2000). For example, in arid regions where long dry periods are followed by short precipitation pulses, water vapor movement is a critical component of the total water flux governing both plant and microbial life (Saito et al., 2006; Or et al., 2007). Saito et al. (2006) found that for fluid flow simulations, coupling liquid water, water vapor, and heat transport is critical for many engineering applications such as the establishment and maintenance of nuclear waste repositories and surface covers for waste disposal sites in arid and semiarid regions. Furthermore, the ability to predict vapor sorption behavior is crucial for remediation of oil, gasoline, and chlorinated chemical contaminations close to storage sites and in cultivated soils. Recently, Solone et al. (2012) showed that erroneous measurement of the SWC can lead to highly inaccurate simulation of the soil water balance.

In addition, soil properties like clay content, specific surface area, solute percolation threshold, or hydrophobicity are intimately linked to sorption kinetics and may be estimated.
from water vapor sorption isotherms (WSI). Soil clay content is certainly the most important soil textural component; it is a fundamental property controlling almost all physical, chemical, and hydrological soil processes. However, traditional methods (e.g., sieving and hydrometer or pipette methods) for measurement of clay content are tedious and expensive. Among others, Waddivira et al. (2012) proposed a regression model to estimate clay content from gravimetric soil water content at a relative humidity (RH) of 0.5 with an acceptable degree of accuracy. Soil specific surface area (SSA) is another important soil property governing processes such as contaminant adsorption, soil aggregation, and microbial attachment and function. The application of polar fluids such as ethylene glycol monoethyl ether (EGME) or methyl glycol to measure SSA provides an estimate close to the actual value due to the ability of polar liquids to penetrate the interlayer space of expandable clay minerals, unlike N2, which only captures external surfaces (Pennell, 2002). However, application of polar liquids requires a difficult-to-establish measurement protocol and raises environmental concerns arising from their disposal. Previous studies (Newman, 1983; Tuller and Or, 2005; Moiseev, 2008; Resurreccion et al., 2011) thus suggest to estimate SSA from WSIs. Bruand and Tessier (2000) showed a strong relationship between SSA and cation exchange capacity (CEC) and suggested estimating properties, such as water repellency (wettability), can be linked to the ability of polar liquids to penetrate the interlayer space of expandable clay minerals, unlike N2, which only captures external surfaces (Pennell, 2002). However, application of polar liquids requires a difficult-to-establish measurement protocol and raises environmental concerns arising from their disposal. Previous studies (Newman, 1983; Tuller and Or, 2005; Moiseev, 2008; Resurreccion et al., 2011) thus suggest to estimate SSA from WSIs.Bruand and Tessier (2000) showed a strong relationship between SSA and cation exchange capacity (CEC) and suggested estimating one from the other. Besides soil processes, all of the above-mentioned soil properties have either a direct or indirect agronomic importance. Soil clay content, SSA, and CEC play a major role in plant nutrient retention, transport, and adsorption. Other soil properties, such as water repellency (wettability), can be linked to the WSI considering sorption kinetics.

The major current experimental limitations for accurate water vapor transport simulations and determination of soil properties from vapor sorption experiments are the time requirements and issues associated with measurement repeatability when generating detailed adsorption or desorption isotherms. If this challenge can be surmounted, numerous opportunities for the advancement of vadose zone research exist. Commonly applied instrumentation for estimating WSIs for soils includes manual, single-point equipment based on chilled-mirror dewpoint theory (Gee et al., 1992), vacuum microbalances, or relative humidity sensors. The WP4-T dewpoint potentiometer (Decagon Devices Inc.) provides relatively fast single-point water potential measurements at predefined soil water contents, but depending on texture, it might require a couple of weeks to ensure adequate sample equilibration before the measurement (Tuller and Or, 2005). A new fully automated instrument, the Vapor Sorption Analyzer (VSA) (Decagon Devices Inc.) that is commonly applied in food industry enables rapid measurements (15–50 h per sample, dependent on texture) of highly detailed adsorption/desorption isotherms for a relative humidity (water activity) range from 0.03 to 0.95. The VSA is a technologically advanced AquaSorp analyzer that was recently employed to determine SSA (Arthur et al., 2013). The rapid, fully automated, and highly repeatable and detailed measurement capabilities of the VSA provide great potential for the advancement of vadose zone research. While a detailed evaluation and description of measurement principles of the VSA are not provided here, the intention of this priority communication is to illustrate potential applications of VSA-generated water vapor sorption isotherms to pertinent issues in vadose zone research.

**Methodology**

**Description of Investigated Soil Samples**

Three sets of soil samples emanating from three geographical regions (Denmark, Sri Lanka, and the United States) were used to illustrate the applicability of the VSA. The first set comprised six Danish soils from two natural texture gradients in Lerbjerg (L1–L6) and Aarup (A1–A5), and two sandy soils from Jyndevad (J1, J2). The second set contained five soils from different regions in Sri Lanka (SL1–SL5), while the third set included six soils from the Long Term Research on Agricultural Systems (LTRAS) project at the University of California, Davis (LT1–LT6), and a Yolo silt loam soil (YS). Soil textural properties and organic carbon (OC) contents for all investigated soils are presented in Table 1. Soil texture measured with a combination of wet sieving and hydrometer methods revealed clay contents ranging from 4 to 46%. Soil OC measured with an organic elemental analyzer ranged from 0.5 to 2.0%. Soil specific surface area (SSA_{EGME}) was measured in triplicate by means of EGME adsorption (Pennell, 2002). Water repellency tests for the two Jyndevad soils were conducted for air- and oven-dry states with the molarity of ethanol drop test (Roy and McGill, 2002; de Jonge et al., 2007).

**Measurement of Water Vapor Sorption Isotherms**

Water adsorption and desorption isotherms were measured with the fully automated VSA. The VSA provides two different isotherm generation modes; the Dynamic Dewpoint Isotherm (DDI) and the Dynamic Vapor Sorption (DVS) methods. Since extensive comparison of the two methods for various soil textures showed no significant difference (Arthur et al., unpublished data), the results presented in this priority communication are based on the DDI method (unless otherwise stated), which is faster and allows for a greater data resolution within a given period of time. In brief, the VSA dries or wets an initially air-dry (or oven-dry) soil sample and automatically measures the water potential with a chilled-mirror dewpoint technique (Gee et al., 1992) while simultaneously recording the sample mass throughout the drying or wetting process with a high-precision magnetic balance. Further details regarding measurement technology and procedures and the theoretical basis for subsequent calculations are presented in Arthur et al. (2013) and Jayanth et al. (2012). The following instrument settings were used for isotherm measurements presented in this study: isotherm measurement mode: DDI; relative humidity (RH)
range: 0.03–0.93; resolution: 0.02 RH; temperature: 25°C; soil mass: about 3.5 g of 2-mm sieved soil. After conclusion of measurements, samples were oven-dried for 48 h to determine the reference oven-dry water contents.

**Applied Models**

In this section we present previously developed models and relationships that were used to estimate SSA, clay content, liquid film thickness, and monolayer coverage based on the measured WSIs.

First, the SSA was estimated based on Eq. [1] from Tuller and Or (2005):

$$\psi = \left( \frac{\rho_w \text{SSA}_{TO}}{\theta_m} \right) \frac{A_{sol}}{6\pi \rho_w g}$$  

where $\psi$ [m H$_2$O] is the matric potential (calculated from RH values with the well-known Kelvin equation), $\theta_m$ [kg kg$^{-1}$] is the gravimetric water content, $\rho_w$ [kg m$^{-3}$] is the density of water, SSA$_{TO}$ [m$^2$ kg$^{-1}$] is the soil specific surface area, $g$ [m s$^{-2}$] is the acceleration due to gravity, and $A_{sol}$ [J] is the Hamaker constant for solid–vapor interactions through the intervening liquid set to $-6 \times 10^{-20}$ J.

Soil clay content was estimated based on a regression equation introduced in Wuddivira et al. (2012):

$$\text{Clay\%} = 1037.5 \times 10^{0.5} \theta_m$$  

where $\theta_m$ is the gravimetric soil water content [kg kg$^{-1}$] at RH = 0.5.

To assess hysteresis and evaluate the effect of oven-drying on the WSIs, the liquid film thickness ($h$, nm) and the number of molecular layers ($m$) of water were estimated at RH = 0.5 (Eq. [3] and Eq. [4]):

$$h = \frac{\theta_m}{\rho_w \text{SSA}_{TO}}$$  

$$\theta_m = \frac{\text{SSA}_{TO}}{\text{SS}_{H2O}} \frac{M_w}{N_A}$$  

where $\text{SS}_{H2O}$ is the surface area occupied by one water molecule (10.8 $\times$ 10$^{-20}$ m$^2$; Livingston (1949)), $M_w$ is the molecular weight of water (18.015 g mol$^{-1}$), and $N_A$ is Avogadro’s number (6.023 $\times$ 10$^{23}$ mol$^{-1}$).

**Results and Discussion**

In subsequent sections, we discuss the applicability of measured WSIs and present links to various eminent vadose zone research topics.

**Vapor Sorption Hysteresis and Water Repellency**

Modeling of physical and biological soil processes is more accurate when hysteresis of the SWC is considered, especially at low saturations where small differences in water contents cause large changes in potential energy (Prunty and Bell, 2007). Nakagawa et al. (2012) reiterated the need to account for hysteresis in numerical models for both two-phase and unsaturated water flows. Hysteresis of the SWC at low saturations for soils of varying texture has been previously considered only in a limited number of studies (Globus and Neusypina, 2006; Prunty and Bell, 2007; Davis et al., 2009; Arthur et al., 2013), most of which did not consider water retained below $-20$ MPa. There is increasing interest in assessing hysteresis at low saturations, primarily due to the introduction of instrumentation that facilitates accurate measurement of WSIs. The WSIs

<table>
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<tr>
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† L, Lerbjerg texture gradient; A, Aarup texture gradient; J, Jyndevad sandy soils, LT, soils from Long Term Research on Agricultural Systems (LTRAS) in UC-Davis; SL, Sri Lankan soils; YS, Yolo Silt Loam soil. OC, organic carbon; SSA$_{EGME}$, soil specific surface area measured with ethylene glycol monoethyl ether; SSA$_{TO}$, soil specific surface area estimated from water vapor sorption isotherms using the Tuller and Or (2005) model. nd, not determined.
for two selected soils (J1 and L6) exhibit significant hysteresis with the effects of oven-drying and soil texture depicted in Fig. 1. As an example, comparison of the water film thickness \( (h) \) at RH = 0.5 for the adsorption and desorption branches \( (H_y = \frac{h_{\text{desorption}}}{h_{\text{adsorption}}}) \) of the WSI can be used as a simple way of quantifying hysteresis, with \( H_y = 1 \) indicating absence of hysteresis. For the air-dry J1 and L6 soils, \( H_y \) at RH = 0.5 was determined as 1.28 and 1.22, respectively. Considering the entire isotherm, Arthur et al. (2013) developed an empirical model with a shape parameter \( N \) for characterizing the WSI. The ratio of \( N \) for adsorption and desorption isotherms was used as a hysteresis/nonsingularity index. These are just two examples of how hysteresis can be quantified. Our aim here is to show that the VSA has potential to provide accurate and highly detailed WSI that can be applied for detailed research on hysteresis/nonsingularity phenomena manifested at the dry-end of the SWC, which are important for numerous ecosystem functions and engineering challenges (e.g., nuclear waste storage) in arid and semiarid environments.

Besides hysteresis, it is important to note the effect of oven-drying on the WSI for coarse-textured soils with high OC content. Oven-drying destroys complexes formed between organic matter and soil mineral components (Koopmans and Groenenberg, 2011). For soils with low clay content and relatively small SSA the effect of oven-drying is expected to be more pronounced. After oven-drying, a remarkable decrease in water sorption at any given RH was observed for the J1 soil, while the WSI of the L6 soil was virtually unaffected by oven-drying (Fig. 1). This is further confirmed by a clear difference in the coverage of water molecules at RH = 0.5, which was 27% and 21% higher for the air-dry WSI compared to the oven-dry WSI for both the adsorption and desorption loops. For the L6 soil, this effect was insignificant (Fig. 1). This indicates that when measuring WSI for low-clay but high-OC soils (clay < 5%; OC > 1.5%), the pretreatment of the samples (air-drying/oven-drying) needs to be taken into account as oven-drying may induce pronounced water repellency.

Water repellency is a global naturally occurring phenomena affecting infiltration (Feng et al., 2001), evaporation, erosion (Cerda and Doerr, 2007), contaminant transport, and preferential flow (Ritsema and Dekker, 1996). Water repellency can be estimated with various standard methods based on contact angle measurements, water drop penetration time, or molarity of an ethanol droplet (de Jonge et al., 2007). Building on the previous discussion about effects of drying on water repellency, the J1 soil was compared to the J2 soil. A previous study (de Jonge et al., 2007) and results from the current study show that the J1 soil is water repellent in the air-dry state, while the J2 is not (Fig. 2). This deduction is based on the water sorption of the two soils: J1, despite having slightly greater clay and OC contents than J2, adsorbed less water at any given RH when compared to J2. After oven-drying, both the J1 and J2 soils exhibit distinct water repellency, as indicated by significantly reduced water sorption for both oven-dry soils when compared to their air-dry state. Based on the shape of isotherms derived from the DVS mode, which shows sorption kinetics and water repellency test results, we envision...
Volatilization and Fate of Soil Contaminants

Volatilization of pesticides and other contaminants (e.g., toluene, trichloroethylene, triallate, trifluralin) in soils is intimately linked to relative humidity and the associated water content (Batterman et al., 1995; Petersen et al., 1996b; Schneider et al., 2013). At low RH (0–0.3), VOCs directly sorb to the mineral surface and are significantly retarded (Batterman et al., 1995; Schneider et al., 2013). However, a sudden increase in soil water content may cause desorption of VOCs, which could potentially lead to large VOC mass fluxes in contaminated areas. Recently, Schneider et al. (2013) showed that increasing RH from 0.6 to 0.85 potentially leads to an eightfold increase in pesticide volatilization rates. Accurate measurement of WSI (as shown here) will facilitate better estimation of this phenomenon and allow identification of the critical RH (and soil water contents) at which volatilization can be reduced to a minimum. This will assist in prediction of the transport and fate of VOCs in soils. For example, Fig. 3 depicts the relationship between the vapor/solid partition coefficient (K_DV) and the relative humidity for the YS soil. The K_DV values and the corresponding soil water content were obtained from published data (Petersen et al., 1995). After measuring the WSI, the relative humidity corresponding to the soil water content was extracted and used in Fig. 3. There is a strong increase in volatilization with increasing moisture content (increasing RH) as evidenced by decreasing K_DV values. The mechanisms responsible for this behavior are discussed in detail in both Petersen et al. (1995) and Schneider et al. (2013). We envision that VSA measurements will provide new opportunities to derive relationships as shown in Fig. 3, which will greatly aid accurate prediction of fate and transport of VOCs and the development of new strategies for remediation of contaminated sites.

Clay Content and Soil Specific Surface Area Estimation

Soil clay content is the single most important property controlling almost all soil processes (hydrological, biological, chemical). Closely linked to clay content is the specific surface area (SSA) (Petersen et al., 1996a). Accurate estimation of both fundamental properties from fast and highly resolved VSA measurements will benefit numerous soil and ecosystem studies. Because SSA_EGME is widely accepted as a measure for the "total" SSA, SSA_EGME was used as the standard for evaluating (Tuller and Or, 2005) VSA-derived SSA (Eq. [1]). Results, similar to those reported by Resurreccion et al. (2011), Arthur et al. (2013), and Maček et al. (2013), show remarkable accuracy of the TO model predictions (Fig. 4a). Further, Maček et al. (2013) reported that the TO model fails for engineering soils with SSA < 5 m²/g, but they pointed out that this limitation can be overcome with more precise water content measurements than they were able to achieve in their study. Although engineering soils were not tested for this priority communication, the experienced accuracy of the VSA most likely will help to overcome this issue.

A comparison of soil clay content estimated from VSA measurements based on the simple regression model proposed by Wuddivira et al. (2012) (Eq.[2]) with the results of the commonly applied combination of wet-sieving and hydrometer methods reveals reasonable accuracy of the VSA estimates with a low RMSE of 4.5% (Fig. 4b). Although soils from three geographical regions, spanning a wide range of textures were considered for this study, the effect of OC on prediction of clay content and surface area cannot be disregarded. High OC contents may undermine the accuracy of the VSA approach for estimating clay content due to the contribution of OC to water sorption, especially for coarse-textured soils (Wäldchen et al., 2012; Chen et al., 2013). This needs to be considered when applying such simple regression models. Further studies are required to evaluate the effects of OC and high silt contents (especially silt 2–20 μm, considering its proximity to delineation of the clay particle fraction) on prediction of clay content and soil specific surface area.

Potential Agronomic Applications

The potential applicability of properties estimated from VSA isotherms, in this case SSA_TOC, to various areas of agricultural research is summarized in Fig. 5. Four soil properties were used for this analysis: clay content, cation exchange capacity (CEC), the Campbell (1974) water retention parameter (θ), and the solute percolation threshold (θ_ch). Soil CEC determines how much plant nutrients

![Graph](image-url)

Fig. 3. Relationship between K_DV values (vapor–solid partition coefficient) and relative humidity for Yolo Silt Loam soil for two organic vapours (toluene and trichloroethylene). K_DV values and corresponding soil water contents were obtained from Petersen et al. (1995) and the relative humidity was estimated based on Vapor Sorption Analyzer (VSA)-measured water sorption isotherms.
are sorbed on soil surfaces. The Campbell $b$ parameter represents the slope of a log-log plot of matric potential and water content for a potential range from $-10$ to $-100$ kPa. The $q_{th}$ parameter, also referred to as the "threshold soil water content for solute diffusion," provides an indication for the amount of water needed before film flow is activated and water starts moving in the liquid phase (Moldrup et al., 2001). Data for soil CEC, $b$, and $q_{th}$, for L1 through L6 were obtained from Olesen et al. (1999). In addition to the widely reported positive correlation between clay content and SSA, CEC, $q_{th}$, and $b$ also show strong correlations with SSA TO.

Soil CEC together with $q_{th}$ represents two critical variables for nutrient retention and transport in agricultural soils. It controls mobility of plant nutrients and pesticides in soils as well as oxygen supply to roots. An increasing solute percolation threshold implies that more water is needed for complete adsorption before interparticle film flow initiates. The strong correlations between CEC, $q_{th}$, and SSA TO (Fig. 5) point to the opportunity of developing models to accurately predict these parameters, rather than measuring them directly. The $b$ parameter, also increasing with increasing SSA TO, is linked to "easily available" water, important for both predictions of gas and solute diffusivities as functions of soil water content, as well as for numerical modeling of water, solute, and gas transport in the vadose zone (Moldrup et al., 2000; Kawamoto et al., 2006; Sommer and Stockle, 2010).

Conclusions

This priority communication introduces advanced fully automated instrumentation (Vapor Sorption Analyzer) for rapid and accurate measurement of water vapor sorption isotherms (WSIs) for soils and discusses potentially wide-ranging applicability for vadose zone research. The relationship between relative humidity (soil water content) and pesticide volatilization was discussed and opportunities to better assess hysteresis and potentially link vapor sorption kinetics to water repellency were illustrated. The WSIs were used to estimate clay content and approximated to obtain soil specific surface area with a high degree of accuracy. Links to the solute percolation threshold and cation exchange capacity were established. The provided overview is by no means complete, but was intended to present some ideas and stimulate new and much needed research on eminent issues in vadose zone science.

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