

Measurement of solid phase permittivity for volcanic soils by time domain reflectometry

K. KAMEYAMA & T. MIYAMOTO

Department of Land and Water Resources, National Institute for Rural Engineering, National Agriculture and Food Research Organization, 2-1-6 Kannondai, Tsukuba, Ibaraki 305-8609, Japan

Summary

Dielectric properties of soils are widely used to estimate their water content. Andisols are unique soils in terms of aggregate structure and show dielectric properties different from other mineral soils. To understand the relationship between the dielectric properties and soil physical properties, multi-phase dielectric mixing models are often used. However, solid phase permittivity (ϵ_s) for Andisols, which is required for calculating the model output, has not been directly measured yet. Therefore, the objectives for this study were to measure ϵ_s for Andisols in Japan. In addition, the measured ϵ_s values were compared with those calculated from the traditional method, which applies two-phase mixing models to estimate ϵ_s values based on effective permittivities of repacked soil samples. The applicability of the traditional method to estimate such values for Andisols is also discussed. The effects of bound water and aggregate structure on measured ϵ_s values were evaluated prior to their measurements. We found that the aggregate structural effects were negligible. However, the amount of bound water caused overestimates of ϵ_s . Four Andisols from the A and B horizons of soils in Memuro Hokkaido (northern part of Japan), from the A horizon of a soil in Tsukuba Ibaraki (central part of Japan), and from the A horizon of a soil in Koshi Kumamoto (southern part of Japan) were used in this study. The ϵ_s values obtained fall between 5.6 and 6.1, and deviated from the estimated ϵ_s values derived from the traditional method. Therefore, the traditional method is probably unsuitable to estimate ϵ_s values for Andisols.

Introduction

Electromagnetic-wave techniques such as time domain reflectometry (TDR) and ground penetrating radar (GPR) are widely used to estimate water content in soils (Noborio, 2001; Férre & Topp, 2002; Huisman *et al.*, 2003; Robinson *et al.*, 2003). These techniques estimate volumetric water content (θ) based on measurements of the effective permittivity of soils (ϵ_{eff}). Hence, knowledge of the θ - ϵ_{eff} relationship is of importance in interpreting ϵ_{eff} measured by these techniques. Topp *et al.* (1980) found the θ - ϵ_{eff} relationship had a unique curve for sandy loam, clay loam and clay soils with a dry bulk density of 1.04–1.44 Mg m⁻³, that is independent of soil texture. They derived an empirical calibration curve:

$$\epsilon_{\text{eff}} = 3.03 + 9.3\theta + 146\theta^2 - 76.7\theta^3. \quad (1)$$

This finding made TDR a popular method for measuring soil water content. However, Topp's calibration curve is not suitable

for organic soils (Topp *et al.*, 1980; Herkelrath *et al.*, 1991; Roth *et al.*, 1992), fine-textured soils (Dasberg & Hopman, 1992), or volcanic soils (Vogeler *et al.*, 1996; Weitz *et al.*, 1997; Tomer *et al.*, 1999; Miyamoto *et al.*, 2001; Regalado *et al.*, 2003; Stenger *et al.*, 2007). In such field soils, a calibration must be performed prior to actual TDR measurements.

Dielectric mixing models are often used both for understanding the relationship between the θ - ϵ_{eff} relationship and soil physical properties, and for predicting calibration curves of specific soils. When modelling a composite material such as soil it is necessary to know the dielectric permittivity (ϵ) of all components (solid phase, water, and air) beforehand. The permittivity of air and water are well documented (Lide, 2006; National Astronomical Observatory, 1990). However, because the permittivity of the solid phase is difficult to measure directly, the permittivity values are often estimated by applying two-phase mixing models for repacked samples. Using this traditional method, Regalado (2004) estimated the solid phase permittivity of volcanic soils. Regalado (2006) recommended using a value close to 15 for volcanic soils. Contrary to this,

Correspondence: T. Miyamoto. E-mail: teruhito@affrc.go.jp

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smaller values have been reported for volcanic rocks such as Pumice (1.7), Rhyolite (4.3–5.5) (Stenger *et al.*, 2007) and for volcanic ash (5.5–6.5) (Adams *et al.*, 1996). These values are also estimated by using the traditional method. Recently, Robinson & Friedman (2003) have proposed a method to measure the permittivity of the solid phase of rocks and soils. This measurement method is termed the ‘immersion method’ because it measures the ϵ_{eff} of samples immersed in fluids with different permittivities. The immersion method has been applied to arid zone soils (Lebron *et al.*, 2004) and clay minerals (Robinson, 2004).

Since the solid phase permittivity for Andisols has not yet been measured directly, the objectives of this study were to measure this by applying the immersion method. In addition, based on the measured data, the applicability of the traditional method to estimate the solid phase permittivity for Andisols is discussed. Andisols are unique soils in terms of aggregate structure, with well-defined and stable intra- and inter-aggregate voids. Therefore, these soils commonly have low natural bulk density, high porosity, relatively large specific surface areas and large water holding capacities. These characteristics contribute to various kinds of physical properties, i.e. water retention, water transmission, thermal conditions (Maeda *et al.*, 1977) and shrinkage characteristics (Poulenard *et al.*, 2002). Therefore, the effects of the aggregate structure and bound water on measured solid phase permittivity for Andisols were evaluated prior to the measurements of this.

Materials and methods

Soil samples

Four Andisols were collected from the A horizon of an experimental field at the National Institute for Rural Engineering in Tsukuba Ibaraki (36°03'N; 140°08'E), A and B horizons of an experimental field at the National Agricultural Research Center for Hokkaido Region in Memuro Hokkaido (42°54'N; 143°03'E), and the A horizon of an experimental field at the National Agricultural Research Center for Kyushu Okinawa Region in Koshi Kumamoto (32°53'N; 130°45'E) (Figure 1). All soil samples were passed through a 2-mm sieve and air-dried

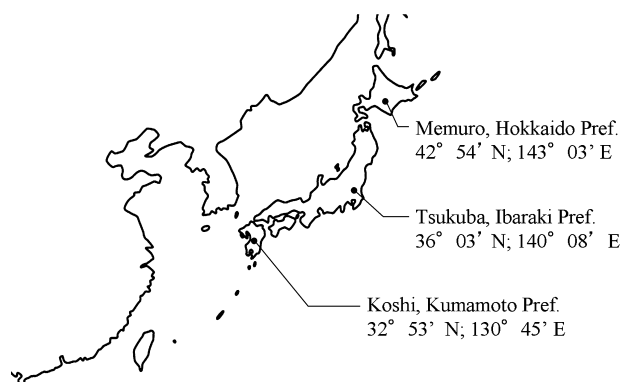


Figure 1 Locations for collection of soil samples.

for 1 month. Particle-size distribution and other components of the soils are shown in Table 1.

To evaluate effects of bound water and the aggregate structure on measured solid phase permittivity, air-dried, oven-dried (110°C for one day and allowed to cool in a desiccator) and crushed oven-dried (crushed with mortar and pestle and oven-dried) soil samples were prepared with the Andisol from Tsukuba. For the other three Andisols, only oven-dried samples were prepared.

Measurement of solid phase permittivity

The immersion method proposed by Robinson & Friedman (2003) was applied to measure solid phase permittivities of the Andisols. The fluids were chosen to obtain values of ϵ_{eff} smaller and greater than the expected ϵ_s value. The relationships between the measured ϵ_{eff} of the soil samples saturated with different fluids and the permittivities of the fluids (ϵ_0) are plotted in Figure 2. The ϵ_{eff} is above the 1:1 line when $\epsilon_s > \epsilon_0$. On the other hand, the ϵ_{eff} is below the 1:1 line when $\epsilon_s < \epsilon_0$. Therefore, the ϵ_s of the soil samples can be obtained from the intersection of the 1:1 line and the fitted curve. In this study, the empirical model of the form $\alpha\epsilon^\beta$ (where α and β are constants) was used as the fitted curve.

The permittivities of the fluids prepared were: air, $\epsilon = 1$; maize oil, $\epsilon = 3.2$; and acetone, $\epsilon = 21$, at 25°C. Acetone and

Table 1 Soil classification, particle-size distribution and other components of the Andisols in this study

Site	Soil classification ^a	Clay/% ^b (<2 µm)	Silt/% ^b (2–20 µm)	Sand/% ^b (>20 µm)	Org.-C /g kg ⁻¹	C/N
Tsukuba (Topsoil)	Typic hydrudand	23.8	21.2	55.0	42.5	11.5
Memuro						
(Topsoil)	Typic hapludand	26.5	11.8	61.7	37.6	13.4
(Subsoil)	Typic hapludand	26.1	29.4	44.6	13.5	9.6
Koshi (Topsoil)	Hydric pachic melanudands	40.5	34.7	24.8	67.2	16.4

^aSoil Survey Staff (2006).

^bDispersion of particles was made by HCl (Nakai, 1997).

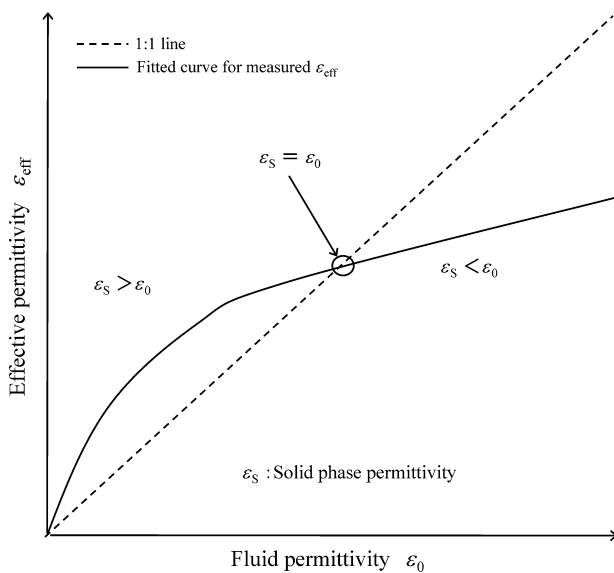


Figure 2 Method to obtain solid phase permittivity.

maize oil are miscible, which allowed us to prepare background fluids with permittivities between 3.2 and 21.

The soil samples were saturated with each fluid as follows:

- 1 Initially the permittivity of the background fluid was measured, and then the fluids were put in a cylindrical container (inside diameter 5 cm; height 7 cm);
- 2 After weighing, the soils were added little by little to the container with gentle tapping to prevent bubbles from forming as much as possible. The soil samples were up to 5.5 cm in height. Surplus fluids were wiped off. The remaining soils were re-weighed to determine the mass of soil poured into the container;
- 3 The same volume fraction of solid was maintained for the subsequent measurements in other fluids to maintain a constant porosity (see Table 2). When the background fluid was air, the soil only was added to the same container.

Table 2 Bulk density, particle density and porosity of soil samples for measurement in this study

Soil samples	Bulk density (S.E.) /Mg m ⁻³	Particle density /Mg m ⁻³	Porosity (S.E.)
Tsukuba			
(Topsoil)			
Oven-dried	0.72 (0.007)	2.61	0.72 (0.003)
Crushed oven-dried	0.89 (0.007)	2.61	0.66 (0.007)
Air-dried	0.72 (0.007)	2.61	0.72 (0.003)
Memuro			
(Topsoil)	0.75 (0.009)	2.52	0.70 (0.004)
(Subsoil)	0.71 (0.009)	2.72	0.74 (0.003)
Koshi			
(Topsoil)	0.75 (0.009)	2.45	0.69 (0.004)

To measure the ϵ_{eff} of the soil samples, a Tektronix 1502B TDR cable tester (Tektronix, Inc., Beaverton, OR, USA) and a custom made TDR probe were used throughout the experiments. The TDR cable tester was connected to a personal computer, which was used to collect and analyse waveforms by means of WinTDR (developed by the Environmental Soil Physics Group at Utah State University, Logan, UT, USA: <http://www.usu.edu/soilphysics/>). The TDR probe was a seven-wire type probe similar to that used by Campbell (1990). The probe was essentially a coaxial transmission line with six outer wires (3 mm in diameter and 50 mm in length) held at ground potential and an inner wire. This probe design results in a well-defined electrical-field volume with nearly no field leakage outside the probe (Campbell, 1990). The probe was inserted vertically into the soil samples. Five waveforms were collected to determine the ϵ_{eff} of the soil samples.

Evaluation of bound water effect using three-phase mixing model

The experimental samples of the air-dried soil were a mixture of three phases: solid particles, water and background fluids. For this problem, Sihvola (1997) presented a simple Maxwell-Garnett-based model for spherical inclusions.

$$\epsilon_{\text{eff}} = \epsilon_0 + 3\epsilon_0 \frac{\left[f_1 \left(\frac{\epsilon_1 - \epsilon_0}{\epsilon_1 + 2\epsilon_0} \right) + f_2 \left(\frac{\epsilon_2 - \epsilon_0}{\epsilon_2 + 2\epsilon_0} \right) \right]}{1 - \left[f_1 \left(\frac{\epsilon_1 - \epsilon_0}{\epsilon_1 + 2\epsilon_0} \right) + f_2 \left(\frac{\epsilon_2 - \epsilon_0}{\epsilon_2 + 2\epsilon_0} \right) \right]}, \quad (2)$$

where ϵ_0 is the permittivity of the background fluids, f_1 is the volumetric fraction of solid with permittivity ϵ_1 , and f_2 is the volumetric fraction of water with permittivity ϵ_2 . Soil water may be divided into free and bound water depending on energy status. The electric polarization of water molecules near the soil surface is limited by the surface charge. As a result, the dielectric permittivity of bound water is much less than that of free water. Many researchers have assumed that the permittivity of bound water is likely to be similar to that of ice, i.e. 3.2 (e.g. Dirksen & Dasberg, 1993; Hilhorst *et al.*, 2001). Therefore, we calculated the ϵ_{eff} from the permittivity of bound water ($\epsilon_2 = 3.2$) or free water ($\epsilon_2 = 80.4$) as water permittivity ϵ_2 .

Estimations using two-phase dielectric mixing modes

The values of ϵ_s are often estimated by applying two-phase dielectric mixing models to the ϵ_{eff} of samples repacked in air. For this method, two kinds of two-phase dielectric mixing models (Lichtenecker's and Looyenga's models) are often used (Olhoeft, 1981; Nelson *et al.*, 1989; Pettinelli *et al.*, 2003; Zheng *et al.*, 2005). In addition, Birchak's model is familiar in soil science (Dirksen & Dasberg, 1993; Miyamoto & Chikushi, 2006). As permittivity of the background was assumed to be 1, the applied mixing models were expressed as follows:

$$\varepsilon_s = \varepsilon_{\text{eff}}^{\frac{1}{1-\phi}} \text{ (Lichtenecker's model),} \quad (3)$$

$$\varepsilon_s = \left(\frac{\varepsilon_{\text{eff}}^{\frac{1}{3}} + f - 1}{f} \right)^3 \text{ (Looyenga's model),} \quad (4)$$

$$\varepsilon_s = \left(\frac{\varepsilon_{\text{eff}}^{\alpha} + f - 1}{f} \right)^{\frac{1}{\alpha}} \text{ (Birchak's model),} \quad (5)$$

where ϕ is the porosity, f is the volumetric fraction of the solid and α in Birchak's model is a parameter concerning the geometrical arrangement of the components. These dielectric mixing models were applied to estimate the ε_s for Andisols and for comparison with the measured ε_s values. Using Birchak's model, $\alpha = 0.5$ was assumed for calculating the ε_s values (Robinson & Friedman, 2003; Regalado, 2004).

Results and Discussion

Effects of aggregate structure and bound water on solid phase permittivity measurement

Figure 3 shows the relationship between ε_0 and ε_{eff} for Andisol from Tsukuba. Plots for oven-dried soils with and without aggregate structure were similar. Therefore, the ε_s values were almost the same, $\varepsilon_s = 5.8$ for the oven-dried soil with aggregate structure and $\varepsilon_s = 5.9$ for the crushed soil. These results indicate that the effect of the aggregate structure is negligible in measuring ε_s for Andisols.

In addition, the plot for air-dried soil was higher than the oven-dried soils (Figure 3), and the ε_s value was 13.5. This value was twice that of the oven-dried soils. The difference

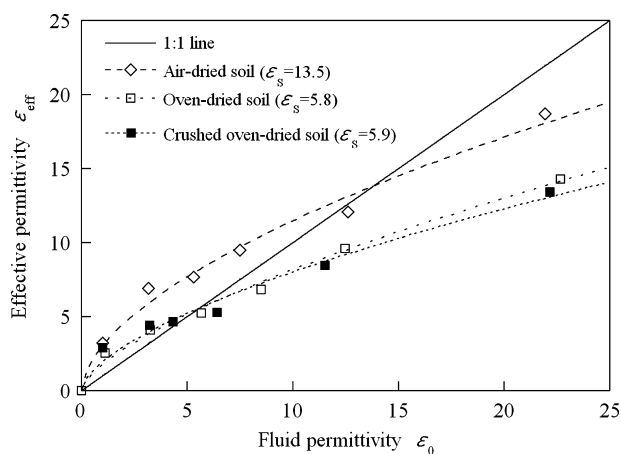


Figure 3 Relationship between fluid permittivity and effective permittivity for the Tsukuba soil samples.

between air-dried and oven-dried soils is likely due to bound water contained in air-dried soil.

The ε_{eff} values calculated from the simple Maxwell-Garnett-based model (Equation (2)) under two different conditions are shown in Table 3. Volumetric water content of the air-dried sample was $0.11 \text{ m}^3 \text{ m}^{-3}$. Therefore, the value was assigned to the variable f_2 in Equation (2). The volumetric fraction of solid (f_1) was calculated from the porosity. The soil samples for measuring dielectric permittivity were prepared immediately after removal from the desiccator. So, rehydration was not considered in this study. When the permittivity value of water was assumed to be 80.4, the calculated ε_{eff} values agreed well with the measured ε_{eff} data for air-dried soil. Contrarily, when the permittivity value of water was assumed to be 3.2, most of the calculated ε_{eff} values were underestimated as compared with the measured ε_{eff} for the five kinds of background fluids. The ε_{eff} values calculated with $\varepsilon_2 = 3.2$ correspond well with the measured ε_{eff} data for the oven-dried soils. These results suggest the water in the three phases (i.e. solid-water-background fluid) is likely to be free water rather than bound water. Therefore, the amount of bound water causes overestimates of the solid-phase permittivity if air-dried soils with large specific surface area are examined. Oven-dried soils should be used to measure ε_s for Andisols by the immersion method.

Measured solid phase permittivity of Andisols

The ε_0 - ε_{eff} relationships for Andisols obtained from two different locations are shown in Figure 4. The measured ε_{eff} values of soils immersed in different fluids gave similar plots. The ε_s values of obtained for the oven-dried soil samples fall within a narrow range: 6.1, 5.7, 5.6 and 5.8 for the topsoil and the subsoil from Memuro, the topsoil from Koshi, and the topsoil from Tsukuba, respectively (Table 4, Figures 3 and 4). Adams *et al.* (1996) measured the complex dielectric permittivity of volcanic ash at frequencies from 4 to 19 GHz. They reported that the real part of the complex permittivity of volcanic ash was 6 ± 0.5 in this frequency range. Zheng *et al.* (2005) measured the dielectric properties of volcanic scoria and basalt at a frequency of 9.37 GHz. They estimated ε_s values for scoria to be within a range of 7.08 to 7.92, and for basalt to be within

Table 3 Comparison between measured effective permittivities and values estimated by Maxwell-Garnett-based model (Tsukuba sample)

	Measured ε_{eff} (Oven-dry)	Measured ε_{eff} (Air-dry)	Model calculated ε_{eff} ($\varepsilon_2 = 80.4$)	Model calculated ε_{eff} ($\varepsilon_2 = 3.2$)
Oil	4.1	6.9	5.1	3.8
Oil 5 : Acetone 1	5.2	7.7	7.1	5.2
Oil 3 : Acetone 1	6.8	9.5	9.1	6.5
Oil 1 : Acetone 1	9.6	12.1	13.1	9.3
Acetone	14.3	18.7	19.7	14.3

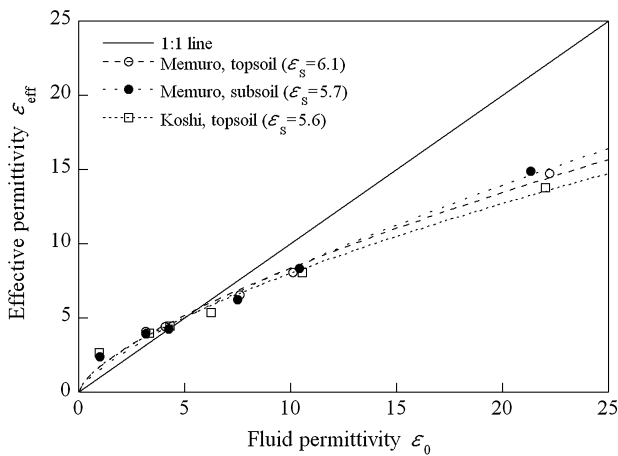


Figure 4 Relationship between fluid permittivity and effective permittivity for the Memuro and Koshi oven-dried soil samples.

a range of 6.86 to 7.86. Our results agree well with these results from materials related to volcanic soils. Therefore, the solid phase permittivity for the Andisols is likely to be correct with a value between 5.6 and 6.1.

Comparison of measured solid phase permittivity with estimations by mixing models

The ϵ_s values measured using the immersion method were compared with the estimated ϵ_s values by applying three different two-phase dielectric mixing models for oven-dried soils (Table 4). Both Looyenga's model and Birchak's model tended to over-estimate the permittivity even though these models can predict the permittivity for granular materials (Robinson & Friedman, 2003), kaolin and illite (Robinson, 2004). Lichtenecker's model over-estimated the permittivity by approximately four times the measured values. Regalado (2004) reported that the ϵ_s values for volcanic soils estimated by Lichtenecker's model and Birchak's model were 13–77 and 7–15, respectively. Our calculated values were within these same ranges (Table 4). These results suggest that Lichtenecker's and Looyenga's models are not suitable for estimating ϵ_s values for volcanic soils, although these models are often used for such. This mismatch is probably because of the geometrical shape of soil

inclusions (Jones & Friedman, 2000). Within the three dielectric mixing models, Birchak models predicted ϵ_s values of the same order of magnitude as the results obtained by the immersion method. We assumed that the fitting parameter of Birchak's model was equal to 0.5 ($\alpha = 0.5$). However, the best-fit α values of Birchak's model were 0.92–1.29. This result suggests that geometrical arrangement of the components is apparently a parallel layering to the electrical field rather than an isotropic arrangement (Roth *et al.*, 1990). The great porosity of the Andisols may contribute to this apparent geometrical arrangement. To obtain reliable values for the solid phase permittivity, the ϵ_{eff} of a repacked sample is often used for estimation. However, as Robinson (2004) pointed out, this method presumes correctness of the model and is thus undesirable if dielectric mixing models are to be rigorously tested. To estimate reliable ϵ_s values for Andisols by using a dielectric mixing model, the model will need further rigorous testing.

From our study, we found the solid phase permittivity for Andisols is not much greater than for quartz ($\epsilon_s = 5$), and is also similar for non-Andisol mineral soils. If the measured ϵ_s values are applied to the dielectric mixing models, the predicted ϵ_{eff} values fall within the range 1.53–2.07. Contrarily, the measured ϵ_{eff} values for oven-dried Andisols repacked in air were 2.39–2.65. These values are greater than the predicted ϵ_{eff} values despite the great porosity. This finding may be a typical dielectric property for Andisols. We speculate that micro-size structures, including the hollow spherule structure of allophanic clays, influence the measured ϵ_{eff} values when the soil samples are repacked in air. This needs to be investigated further.

Conclusions

Measurements of the solid phase permittivity of four Andisols by using the immersion method are presented. In addition, the measured values of ϵ_s were compared with calculated ϵ_s from the traditional method. The results are summarized as follows:

- 1 The ϵ_s values obtained for the oven-dried soil samples fall within a narrow range: 6.1, 5.7, 5.6 and 5.8 for the topsoil and the subsoil from Memuro, the topsoil from Koshi, and the topsoil from Tsukuba, respectively.
- 2 Applying the immersion method for Andisols, the aggregate structural effects were negligible. However, the amount of

Table 4 Measured effective permittivities of the oven-dried soil/air systems, estimated values by various dielectric mixing models and measured permittivities of solid phase for the oven-dried Andisols

	Measured ϵ_{eff}	Lichtenecker	Looyenga	Birchak	Measured ϵ_s
Tsukuba (Topsoil)	2.54	28.8	12.4	9.9	5.8
Memuro					
(Topsoil)	2.40	20.7	10.3	8.4	6.1
(Subsoil)	2.39	34.3	13.3	10.3	5.7
Koshi (Topsoil)	2.65	21.1	10.7	8.8	5.6

bound water caused overestimates of ϵ_s . Therefore, oven-dried soils should be used to measure ϵ_s for Andisols by the immersion method.

3 Birchak's model predicts ϵ_s values of the same order of magnitude as the results obtained by the immersion method. However, Lichtenecker's and Looyenga's models tend to overestimate the ϵ_s values.

4 We speculate that micro-size structures, including the hollow spherule structure of allophanic clays, influence the calculated ϵ_s values when the traditional method is applied.

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