Time domain reflectometry measured moisture content of sewage sludge compost across temperatures

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ABSTRACT

Time domain reflectometry (TDR) is a prospective measurement technology for moisture content of sewage sludge composting material; however, a significant dependence upon temperature has been observed. The objective of this study was to assess the impacts of temperature upon moisture content measurement and determine if TDR could be used to monitor moisture content in sewage sludge compost across a range of temperatures. We also investigated the combined effects of temperature and conductivity on moisture content measurement. The results revealed that the moisture content of composting material could be determined by TDR using coated probes, even when the measured material had a moisture content of 0.581 cm⁻³, temperature of 70 °C and conductivity of 4.32 mS cm⁻¹. TDR probes were calibrated as a function of dielectric properties that included temperature effects. When the bulk temperature varied from 20 °C to 70 °C, composting material with 0.10–0.70 cm⁻³ moisture content could be measured by TDR using coated probes, and calibrations based on different temperatures minimized the errors.

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1. Introduction

Measurement of moisture content (MC) is a key necessity for management of sewage sludge (SS) composting. The gravimetric method is usually employed to measure the MC of the composting material. The gravimetric method is based on a simple theory and has been widely used as a reference method (Larossa Rodríguez et al., 1999; Bittelli et al., 2008). However, during sludge composting, sampling for the gravimetric method is laborious and the drying time consuming; therefore, the MC cannot be determined in real time. Contrary to the gravimetric method, a rapid and accurate in situ approach known as time domain reflectometry (TDR) enables non-destructive monitoring of MC. TDR has the potential for use in moisture management during sludge composting (Yue, 2008; Černý, 2009).

TDR is based on determination of the dielectric constant of the material being analyzed, which is correlated with the MC of the material. Indeed, the MC can be determined from the dielectric constant (Noborio, 2001); however, the results will be affected by characteristics such as electrical conductivity, temperature, bulk density and composition (Wraith and Or, 1999; Moret-Fernández et al., 2009), all of which impact determination of the dielectric constant, consequently affecting the MC. Among these factors, the effects of electrical conductivity can be minimized by using coated probes (Robinson et al., 2003; McIsaac, 2010). The bulk density has a slight effect on the MC measurement of composting material. During SS composting, the bulk density usually varies from 580 kg m⁻³ to 886 kg m⁻³ (Yue et al., 2008; Chen et al., 2011), a range in which a unified calibration can be applied robustly ($R^2 = 0.98$) and the MC values determined at different densities are within 3.8% errors (Yue, 2008). The temperature of the sludge bulk can exceed 55 °C during composting, which has a strong effect on measurement of the dielectric constant.

Temperature affects the dielectric constant by factors such as bulk conductivity and the polarization of free and bound water (Calvet, 1975; Evett et al., 2012). The dielectric constant of the composting material increases with increased temperature (Pepin et al., 1995). Schwartz et al. (2009a) demonstrated that the response of apparent dielectric constant to temperature depends on the interdependent effects of the relaxation times for free and bound water as well as the magnitude of the bulk electrical conductivity. The bulk conductivity shows strong temperature dependence and the chief charge carriers responsible for the bulk conductivity appeared to be the movement of protons (Hunt et al., 2006). When temperature impacts the bulk electrical conductivity, it influences the dielectric constant indirectly (Persson and Berndtsson, 1998; Sun and Young, 2001).

During SS composting, temperature varies so dramatically that the temperature fluctuations have significant effects on the MC...

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measurement (Schwartz et al., 2009b, 2010); however, the effects and corresponding calibrations of compost piles at different temperatures have not been investigated. This lack has limited the application of TDR during the process of SS composting. To improve the accuracy of MC measurement of SS composting material by TDR, the effects of temperature should be considered and calibrations should be performed.

The purpose of this study was to (i) determine the effects of temperature and electrical conductivity on the measurement of SS’s MC; and (ii) develop calibration equations for moisture content as a function of dielectric properties that include temperature effects.

2. Measurement theory

TDR determines the apparent dielectric constant $K_a$ of sludge composting material by measuring the travel time of a high frequency electromagnetic wave through a wave-guide probe inserted into the material. The transmitting velocity of the wave is then calculated and used to determine the dielectric constant of the material. Finally, the MC $\theta$ is calculated based on the relationship between $K_a$ and $\theta$ (Noborio, 2001).

The velocities of electromagnetic waves vary in different materials, and can be calculated as follows:

$$v = \frac{2L}{t}$$  \hspace{1cm} (1)

where $v$ is the propagating velocity of the electromagnetic wave (m s$^{-1}$); $L$ is the length of the probe (m); and $t$ is the travel time (s), which can be obtained according to the first and second reflections (see Fig. 1).

According to electromagnetic theory, the velocity of an electromagnetic wave in material is determined by the dielectric constant and magnetic susceptibility of this material:

$$v = \frac{c}{\sqrt{\mu_r \varepsilon_r}}$$  \hspace{1cm} (2)

where $c$ is the velocity of the electromagnetic wave in free space (m s$^{-1}$); $\varepsilon_r$ is the relative dielectric constant; and $\mu_r$ is the relative magnetic susceptibility (usually being 1). (1) and (2) can be rearranged as follows:

$$\varepsilon_r = \left( \frac{ct}{2L} \right)^2$$  \hspace{1cm} (3)

During determination of the dielectric constant, the apparent dielectric constant $K_a$ is measured based on calculation from the relative dielectric constant $\varepsilon_r$. Therefore, $K_a$ is obtained based on the travel time $t$ (Černý, 2009).

3. Materials and methods

3.1. Experimental materials

SS was collected from the municipal wastewater treatment plant of Qinhuangdao, China. Sawdust (SD) was acquired from wood working factories in the same city. Bio-dried product (BP) was collected from the Lvgang Municipal Sewage Sludge Treatment Plant (Qinhuangdao, China) (Cai et al., 2010). SD and BP were used as bulking agents for composting. The MC values of SS, SD and BP were 0.856 cm$^3$ cm$^{-3}$, 0.039 cm$^3$ cm$^{-3}$ and 0.200 cm$^3$ cm$^{-3}$, respectively. The bulk densities of SS, SD and BP were 1.04 g cm$^{-3}$, 0.19 g cm$^{-3}$ and 0.49 g cm$^{-3}$, respectively.

3.2. Experimental procedures

During investigation of the temperature effects on the MC measurement by TDR, three raw materials were mixed until the MC of the mixture reached 0.581 cm$^3$ cm$^{-3}$. This value was selected because it is recommended that the initial MC of the composting material be less than 0.60 cm$^3$ cm$^{-3}$ during sludge composting. Seventeen constant temperatures present in the sludge composting material were set based on variations of temperature during the process of sludge composting. Because the peak temperature of composting bulk could reach 70 °C, material with an MC of 0.581 cm$^3$ cm$^{-3}$ was dried at 20 °C, 30 °C, 40 °C, 50 °C, 60 °C and 70 °C. At each temperature, $\theta$, $K_a$ and the electrical conductivity ($\sigma$) of the tested materials were measured.

The reference value $\theta$ of the composting material was determined by the gravimetric method after drying the material at 75 °C in an oven until it reached a constant weight (Rynk, 2000). To determine the volumetric MC, gravimetric MC was multiplied by the bulk density of the material. Composting material was then sampled by a ring sampler, weighed, and its bulk density was calculated.

During measurement of $K_a$, the signal was launched and the waveform was recorded by TDR (Soil Moisture Equipment Corp., Santa Barbara, CA, United States). For this measurement, probes were made of three stainless steel rods with a length of 20 cm and a diameter of 3 mm. The middle rod was coated with a polymer material to reduce interference caused by conductivity (Mortet-Fernandez et al., 2009). Waveforms were analyzed using the dual-tangent method provided by the WinTrase software (Soil Moisture Equipment Corp., Santa Barbara, CA, United States). The measured $K_a$ was smaller than the true value because a coated probe was used for the experiment. $\sigma$ was measured in situ using a conductivity meter (Hanna Instruments Corp., Padova, Italy).

During sludge composting, the mixing ratio of SS, BP and SD was set at a volumetric ratio of 3:2:1 (SS: BP: SD) to give an initial moisture and free air space that was appropriate for microbial fermentation (Adhikari et al., 2009; Cai et al., 2010; Chen et al., 2011). The process of sludge composting was conducted using CTB auto-control technology (Control Technology of Bio-composting). The period of sludge composting was 20 days. As shown in Fig. 2, TDR probes and a temperature sensor were inserted into the bulk at a depth of 0.6 m (from top to bottom). Bulk temperature was monitored in real time by a PT100 temperature sensor throughout the process. The MC of the composting material was measured by
4. Results and discussion

4.1. Temperature effects on dielectric constant measurement

Composting material with a 0.581 cm$^3$cm$^{-3}$ MC was tested at different temperatures and the corresponding waveform of TDR was analyzed (Fig. 3). The angle of the second reflection became smooth and its reflection point was delayed at higher temperatures, leading to prolonged travel time $t$ and an overestimated $K_d$ (Fig. 3). Nevertheless, even when the temperature was 70 $^\circ$C, the second reflection angle of the waveform still existed, and the second reflection point could be judged. In the wide temperature range of composting bulk material, $K_d$ could be measured by TDR using coated probes.

As shown in Fig. 4, the value of $K_d$ for material with the same MC increased when the temperature was higher. Temperature had a greater effect on the dielectric constant for material with an MC larger than 0.393 cm$^3$cm$^{-3}$. Temperature had a remarkable effect on MC measurement, even when materials had the same MC, suggesting that the effects of temperature on the MC measurements by TDR should be taken into account and individual calibration of the $K_d$-$\theta$ relationship of composting material should be conducted at different temperatures during the MC measurement of composting material.

4.2. Combined effects of temperature and conductivity

Temperature could change the conductivity of materials that attenuate the TDR signal, thereby affecting the measurement of $K_d$. Accordingly, $K_d$ could be affected by the combined effects of temperature and conductivity. As shown in Fig. 5, the conductivity of material with the same MC increased when the temperature was higher; however, the conductivity was not larger than 5 mS cm$^{-1}$ at the highest temperature. Based on these findings, the material could still be measured by TDR using coated probes.

The waveforms of measurements taken at 70 $^\circ$C are shown in Fig. 6. The signal was attenuated and the second reflection angle of the waveform became more flat in response to the combined effects of high temperature and conductivity. However, TDR conducted using coated probes reduced the combined effects of these factors. As shown in Fig. 6, when a material had an MC of 0.581 cm$^3$cm$^{-3}$, temperature of 70 $^\circ$C and conductivity of 4.32 mS cm$^{-1}$, a second reflection was still present and could be used to determine the dielectric constant. Therefore, the combined

![Fig. 2. The structure of the sewage sludge composting experiment (unit: m).](image)

![Fig. 3. Measurement waveforms of sludge composting material with a 0.581 cm$^3$cm$^{-3}$ MC at different temperatures.](image)

![Fig. 4. Apparent dielectric constant of sludge composting material of different MCs at different temperatures. Profiles with different symbols indicate materials with different MCs. Error bars show standard deviations of means ($n = 15$).](image)
effects of high temperature and conductivity will not interrupt the MC measurement, even when a sludge composting material with a high MC in the thermophilic phase is being analyzed.

4.3. Interpretations of temperature effects on measurements

The temperature effects on MC measurements during the process of SS composting are caused by the interplay among the static dielectric constant of free water, the volumetric fraction of bound water, and the bulk electrical conductivity (Schwartz et al., 2009a). There are at least two dielectric relaxations for water associated with smectites (Calvet, 1975). In bound water, the molecules exhibit an increase in relaxation strength at increased temperatures. Electrical conductivity contributions to losses are also strongly tied to the dielectric constant (Schwartz et al., 2009a). Tirolroij et al. (2009) demonstrated that an increase in temperature results in the conversion of bound water to water exhibiting bulk-like behavior. Changes in the properties of bound water affected the aggregate response of the measurement of dielectric constant. As shown in Fig. 4, temperature changed the dielectric constant of the sludge composting material. The “release” of bound water with increasing temperature (Vesilind, 1994; Wraith and Or, 1999) introduced an evident change in the dielectric constant, even when the MC of the material was constant.

4.4. Application of TDR after calibration during sludge composting

Fig. 7 shows the relationship between the $K_r$ and $\theta$ of sludge composting material at 20°C, 30°C, 40°C, 50°C, 60°C, and 70°C. The relationships fitted by 3rd-order polynomials are listed in Fig. 7. The fitting results showed good correlations with $R^2$ values of 0.996, 0.996, 0.993, 0.995, 0.996, and 0.994, respectively. Sludge composting and MC measurement were conducted simultaneously. The thermophilic phase of sludge composting requires a long time (15–17 days of a 20 day process). In this experiment, the thermophilic phase of sludge composting bulk material started from day 2 and ended on day 17 (when the temperature of the composting bulk had decreased to below 40°C, as shown in Fig. 8). These findings indicated that the mesophilic phase was short. During MC measurement in the thermophilic phase, the $K_r-\theta$ relationship at a temperature gradient close to the actual bulk temperature was selected for calibration of the TDR probes. Based on the rapid self-heating of the bulk material (40°C on day 2), as well as the operation convenience and measurement accuracy, one general $K_r-\theta$ relationship at 20°C was employed for composting material during the mesophilic phase, i.e., the $K_r-\theta$ relationship at 20°C was employed on day 1 (when the bulk temperature was 22.5°C) and on day 18 to day 20 (when the bulk temperature ranged from 27.4°C to 21.9°C).

Bulk temperature changed during the process of SS composting. A unified equation including water variation with both dielectric and temperature was developed:

$$
\theta = 2.80 \times 10^{-2} + 1.44 \times 10^{-2}T + 4.48 \times 10^{-2}K_d + 3.21 \times 10^{-5}T^2 - 3.33 \times 10^{-5}T \cdot K_d - 9.34 \times 10^{-4}K^2_d - 4.54 \times 10^{-7}T^3 - 3.99 \times 10^{-7}T^2 \cdot K_d + 7.81 \times 10^{-6}T \cdot K^2_d + 5.45 \times 10^{-6}K^3_d$$

$$
(R^2 = 0.987) \tag{5}
$$

where MC of the composting material at any temperature could be determined. During engineering of SS composting, the initial and final MC values are the primary objects of interest; therefore, the individual calibration equation at a specific temperature is
Turning models worked for the measurements during SS composting. The MC of the composting material on each day was obtained using calibrated TDR probes and the gravimetric method (see Table 1). The results revealed that there was no significant difference ($P > 0.05$) between the MC values determined by these two methods when using paired samples $T$ test. When the values determined by TDR were calibrated with those obtained by the gravimetric method, the relative error was $0.92 \pm 0.72\%$, indicating that the values determined by TDR were highly accurate. The standard deviation (SD) of values determined by TDR was smaller than that of values determined by the gravimetric method (see Table 1). The larger SD determined by the gravimetric method was due to the errors produced during sampling and drying of the tested material, which could be avoided during in situ measurement by TDR. Therefore, the results of TDR after calibration at different temperatures had high accuracy and good stability, indicating that the calibration models worked for the measurements during SS composting.

### Table 1

<table>
<thead>
<tr>
<th>Day</th>
<th>TDR method (cm$^3$ cm$^{-1}$)</th>
<th>Gravimetric method (cm$^3$ cm$^{-1}$)</th>
</tr>
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<tr>
<td></td>
<td>Fitted by individual equations</td>
<td>Fitted by a unified equation</td>
</tr>
<tr>
<td>1</td>
<td>$0.542 \pm 0.001$</td>
<td>$0.545 \pm 0.001$</td>
</tr>
<tr>
<td>2</td>
<td>$0.538 \pm 0.003$</td>
<td>$0.537 \pm 0.005$</td>
</tr>
<tr>
<td>3</td>
<td>$0.533 \pm 0.001$</td>
<td>$0.534 \pm 0.002$</td>
</tr>
<tr>
<td>4</td>
<td>$0.535 \pm 0.003$</td>
<td>$0.535 \pm 0.004$</td>
</tr>
<tr>
<td>5</td>
<td>$0.499 \pm 0.002$</td>
<td>$0.495 \pm 0.002$</td>
</tr>
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<td>$0.471 \pm 0.002$</td>
<td>$0.470 \pm 0.003$</td>
</tr>
<tr>
<td>7</td>
<td>$0.458 \pm 0.002$</td>
<td>$0.459 \pm 0.003$</td>
</tr>
<tr>
<td>8</td>
<td>$0.454 \pm 0.003$</td>
<td>$0.453 \pm 0.004$</td>
</tr>
<tr>
<td>9</td>
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<td>$0.452 \pm 0.001$</td>
</tr>
<tr>
<td>10</td>
<td>$0.446 \pm 0.0001$</td>
<td>$0.446 \pm 0.0005$</td>
</tr>
<tr>
<td>11</td>
<td>$0.451 \pm 0.0002$</td>
<td>$0.452 \pm 0.001$</td>
</tr>
<tr>
<td>12</td>
<td>$0.447 \pm 0.0003$</td>
<td>$0.450 \pm 0.001$</td>
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<tr>
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<td>$0.436 \pm 0.001$</td>
<td>$0.436 \pm 0.001$</td>
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<td>$0.425 \pm 0.001$</td>
<td>$0.425 \pm 0.002$</td>
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<tr>
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<td>$0.414 \pm 0.001$</td>
<td>$0.412 \pm 0.001$</td>
</tr>
<tr>
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<td>$0.391 \pm 0.003$</td>
<td>$0.392 \pm 0.004$</td>
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<tr>
<td>17</td>
<td>$0.387 \pm 0.0004$</td>
<td>$0.383 \pm 0.003$</td>
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</tr>
<tr>
<td>20</td>
<td>$0.352 \pm 0.001$</td>
<td>$0.354 \pm 0.001$</td>
</tr>
</tbody>
</table>

Results are the means ± SD. Replicates for the TDR and gravimetric method were 15 and 5, respectively.

### 5. Conclusions

The measured $K_p$ of sludge composting materials with the same MC increased with temperature. For material with different MCs, temperature had a greater effect on the measured MC when the MC was higher. The conductivity of the composting material varied moderately at different temperatures, and had no effect on measurements by TDR using coated probes.

Based on these results, it is recommended that the $K_p-\theta$ relationship of composting material be individually calibrated at different temperatures to improve the measurement accuracy. MC determined by calibrated TDR probes had high accuracy and good stability when compared with that determined by the gravimetric method.

### Acknowledgments

This project was financially supported by the National High-tech Research and Development Program of China (863 Program) (Nos. 2009AA064703 and 2008AA062402) and the National Water Pollution Control and Management Technology Major Project of China (No. 2009ZX07318-008-007). We gratefully acknowledge the helpful suggestions of David Moret-Fernández during the initial stage of this research, as well as the dedicated support of Okoli Peter Chukwunonso and Shaohua Wang. We also thank Associate Editor Uta Krogmann and three anonymous reviewers for their constructive comments.

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