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TECHNICAL NOTE

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Effects of Oven Temperature and Addition of Ethanol on Measurement of Water Content and Specific Gravity of Cement-Stabilized Soft Clay

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ABSTRACT

Water content and specific gravity are two fundamental soil properties. The water content is usually measured by the oven-drying method, and the dried soil is used for specific gravity determination of the soil. However, for cement-stabilized soils, high oven temperature accelerates the cement hydration rate and causes dehydration of some clay minerals, which may affect the measurement accuracy of water content and soil specific gravity. Hence, researchers have proposed the use of ethanol to terminate cement hydration reactions and a low oven temperature to reduce the dehydration of hydration products. To verify the effectiveness, this experimental study investigated the effects of oven temperature (60°C and 110°C) and the addition of ethanol on the measurements of both water content and specific gravity of cement-stabilized soft clay. These measurements are compared with those measured with the freeze-drying method, which is termed as a control measurement. The test results indicated that the effect of ethanol on water content measurement was insignificant. For 7-day stabilized clay, oven drying at 110°C achieved lower errors (3.06–3.63 %) than the oven-drying method at 60°C (7.36–9.44 %). For 180-day cement-stabilized clay, all the oven-drying methods had similar measurement errors (4.02–5.58 %). The effect of drying method on the specific gravity measurement was insignificant, with the percentage of measurement error ranging from 0.19 to 2.17 %.

Keywords

water content, specific gravity, stabilized clay, oven temperature, ethanol

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Introduction

Water content, defined as the mass of water over the mass of solid, is a fundamental soil property. Generally, there are three types of water in the soil, including free water (i.e., water that is not associated with soil particles and that can be evaporated without external energy), bound water (i.e., water physically held by soil particles because of attraction forces), and hydration water (i.e., chemically bound water within particle structure as a part of soil minerals) (Bouyoucos 1921; Bayer 1956; Mitchell and Soga 2005; Smith and Vesilind 1995). Hence, only the free and bound water are considered as the “water” content of soil in geotechnical engineering, and it is ideal to remove free and bound water without changing the hydration water when measuring the water content. The oven-drying method is widely used to determine the water content of the soil in geotechnical engineering, and it has benefits of simplicity and low cost. ASTM D2216-19, *Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*, indicates a standard method for measuring the water content of soil with oven-drying at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$, and it also indicates that the oven temperature of 60°C is desirable for drying materials that dehydrate at less than 110°C .

For cement-stabilized soil, the water content does not only affect the engineering performance, e.g., permeability, compressibility, and strength but it is also essential for quantifying other properties including dry density, void ratio, and degree of saturation. Therefore, it is important to measure the water content of cement-stabilized soil consistently and accurately. Currently, there is no specified ASTM standard for determining the water content of cement-stabilized soil. Some ASTM standards (ASTM D559/D559M-15, *Standard Test Methods for Wetting and Drying Compacted Soil-Cement Mixtures*; ASTM D560/D560M-16, *Standard Test Methods for Freezing and Thawing Compacted Soil-Cement Mixtures*; ASTM D558/D558M-19, *Standard Test Methods for Moisture-Density (Unit Weight) Relations of Soil-Cement Mixtures*) indicate that the water content of cement-stabilized soil can be determined according to ASTM D2216-19 without specifying the appropriate oven temperature for the measurement. For cement-stabilized soil, it is necessary to consider the effects of cement hydration reactions and hydration products during water content measurement. The crystalline water in hydration products, e.g., calcium silicate hydrate (CSH) and ettringite, belongs to hydration water, and a high oven temperature of 110°C can not only change the hydration products (e.g., thermal dehydration of ettringite) but also accelerate the cement hydration rate, i.e., the water content is changing during measurement (Lu, Tan, and Phoon 2012; Zhang et al. 2014). For cement-based materials, the organic solvent exchange technique is often applied to stop cement hydration by exchanging water in pores without removing the water in hydration products (Zhang and Scherer 2011; Scrivener, Snellings, and Lothenbach 2016). For cement-stabilized soil, previous researchers, including Yi et al. (2016) and Yin et al. (2018), used ethanol to terminate hydration reactions and a low oven temperature (40°C – 70°C) to reduce the removal of crystalline water in hydration products. In both studies, the sieved stabilized soil was weighed and immersed in the ethanol for a period of time (≥ 1 day) and then moved to the oven for drying. However, the effectiveness of this method has not been verified in water content measurement of cement-stabilized soil.

Specific gravity is another fundamental soil property, and the dry soil is needed for the measurement. Generally, oven-dried soil is used for the measurement of specific gravity according to ASTM standards (ASTM D5550-14, *Standard Test Method for Specific Gravity of Soil Solids by Gas Pycnometer*; ASTM D854-14, *Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer*). Hence, similar to that of water content, the oven temperature and addition of ethanol may affect the specific gravity of cement-stabilized soil. To the best of the authors’ knowledge, there is no study on the effects of oven temperature and addition of ethanol on the measurement of water content and specific gravity of cement-stabilized soil. Hence, this study attempts to fill the gap. The following sections describe the experimental program and tasks followed to address the effects of oven temperature and ethanol treatments to measure the basic soil properties.

Materials and Laboratory Experiments

MATERIALS AND SPECIMEN PREPARATION

A Singapore soft marine clay was used in this study. The clay had plastic and liquid limits of 25.2 and 58.9 %, respectively, determined according to ASTM D4318, *Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils*, and organic content of 0.89 %, determined according to ASTM D2974, *Standard Test Methods for Determining the Water (Moisture) Content, Ash Content, and Organic Material of Peat and Other Organic Soils*. An ordinary portland cement obtained from Engro Co. Ltd., Singapore, was used to stabilize the clay. The chemical composition of clay and cement was tested by an X-ray fluorescence spectrometer as shown in Table 1.

The wet cement-clay mixture was designed according to commonly used values of cement content and water/cement mass ratio for wet deep-mixing applications. The cement content is defined as the cement/dry soil mass ratio. The total amount of water in the mixture was constituted by two parts: (1) water in a cement slurry quantified based on water/cement ratio and cement content and (2) water in the clay quantified based on in situ water content. The cement content of 30 % and water/cement ratio of 1.0 were used in this study based on common practical values for deep cement mixing in soft clays in Singapore (Lee et al. 2005; Lee, Lee, and Dasari 2006). Considering the in situ water content of 50–70 % of the clay, a water content of 60 % was used in the laboratory. Therefore, the initial water content in the wet cement-clay mixture before hydration (w_i) could be calculated as

$$w_i = \frac{M_{w1} + M_{w2}}{M_s + M_c} \times 100 \% \quad (1)$$

where M_{w1} is the mass of water in cement slurry, M_{w2} is the mass of water in clay, M_s is the mass of dry clay, and M_c is the mass of cement. Referring to equation (1), the initial water content of the mixture before cement hydration was 69.2 % calculated based on the clay water content (60 %), cement content (30 %), and water/cement ratio (1.0).

EXPERIMENTAL PROCEDURE

The clay was first air-dried, grounded using a mechanical grinder, and then sieved with a sieve of an aperture size of 2.36 mm (ASTM No. 8 sieve). The sieved clay was oven-dried at a temperature of 110°C for 24 h to ensure the dry state of the soil. The cement and dry clay, weighted based on the test program, were mixed and homogenized for 5 min in a laboratory mixer at 60 rpm. The weighted water was added and mixed for another 5 min to form a slurry mixture. The prepared mixture was then filled in cylindrical molds with 50 mm of diameter and 100 mm of height in three layers, according to specifications proposed by Kitazume and Terashi (2013).

After each addition of the mixture, the mold was manually tapped against a laboratory table 50 times to remove air bubbles (Kitazume and Terashi 2013). Each specimen within the mold was sealed in an airtight plastic bag to minimize the evaporation and placed in a moisture room with the constant relative humidity of 95 ± 3 % and the temperature of $23^\circ\text{C} \pm 2^\circ\text{C}$. The cement-stabilized clay specimens were cured for 7 and 180 days, representing relatively short-term and long-term conditions, respectively. After curing for 7 days, all specimens were

TABLE 1

Chemical composition (by percent weight) of soft clay and cement

Material	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	K ₂ O	TiO ₂	MnO	Na ₂ O	LOI
Clay	1.80	64.60	20.39	5.79	0.80	1.47	2.21	1.17	0.05	0.84	0.89
Cement	62.13	20.77	3.66	2.93	1.76	4.11	0.35	0.69	0.06	0.37	3.16

Note: LOI = loss on ignition.

demolded. The rest of the specimens were sealed in plastic bags again to continue curing up to 180 days. After reaching designated curing periods (7 and 180 days), the crushed samples were further hammered to finer particles within airtight bags and then quickly sieved (<10 s) through the sieve of 2.36 mm for water content measurement.

Using organic solvents for arresting hydration of cement has been studied for decades (Taylor and Turner 1987; Thomas 1989; Beaudoin et al. 2000; Zhang and Scherer 2011). Ideal physical properties of a solvent include small molecule size, low boiling point, capability of being miscible with water, high diffusivity in water, and low surface tension (Zhang and Scherer 2011). Commonly used organic solvents include acetone, ethanol, methanol, and isopropanol (Scrivener, Snellings, and Lothenbach 2016). Methanol shows a disadvantage in strongly interacting with hydration products, especially ettringite (Taylor and Turner 1987; Khoshnazar et al. 2013). Beaudoin et al. (1998, 2000) reported that there would be chemical reactions between solvents (acetone, methanol, and isopropanol) and calcium hydroxide. Hansen and Gran (1996) found that there was no sign of chemical interaction between ethanol and cement paste detected by carbon-13 nuclear magnetic resonance spectroscopy. Therefore, the ethanol was used in this study for stopping hydration of cement in the stabilized soil.

Five drying methods were used in this study considering the effects of oven temperature and ethanol on the water content measurement of cement-stabilized clay, which include oven-drying at 110°C (OD110), oven-drying at 110°C with ethanol (ED110), oven-drying at 60°C (OD60), oven-drying at 60°C with ethanol (ED60), and freeze-drying (FD). OD110 and OD60 indicate sieved samples directly placed to the ovens at 110°C and 60°C, respectively. ED110 and ED60 denote sieved samples immersed in ethanol for 24 h before transferring to the ovens at 110°C and 60°C, respectively. The mass ratio of ethanol liquid/cement-stabilized soil was 1.5/1 in this study and equivalent to an ethanol liquid/cement volume ratio of ~22/1, which is larger than the minimum liquid/solid volume ratio of 20/1 recommended by Kowalczyk, Gajewicz, and McDonald (2014). Considering the diffusivity of solvent and dimensions of samples, Zhang and Scherer (2011) estimated a soaking time of 18 h is required to replace 99.9 % water with organic solvent in a granule of cement paste with a diameter of 2 mm. Therefore, the soaking time in this study was set as 24 h for sieved cement-stabilized soil samples with a diameter of less than 2.36 mm. The FD method is widely used in cement research, which can dry cement samples without changing the hydration water (Konecny and Naqvi 1993; Gallé 2001). Compared with oven-drying and solvent exchange methods, the FD method shows the advantage of preserving mineral composition (Zhang and Scherer 2011). In this study, the FD was used as a control, for which the sieved samples were directly placed to a freeze-dryer (Martin Christ Alpha 1-2 LD plus). The freeze-drying process contains two consecutive stages. Samples were first frozen with a temperature of -60°C for 24 h and were then kept at this temperature and a vacuum pressure of 6 Pa for another 24 h, referring to Knapen and Van Gemert (2009). Five replicate samples were taken for each drying method. All samples were dried for 24 h and then weighted. Additional measurements (at least two) were conducted for each period of 24 h to ensure that the samples reached the constant mass.

According to ASTM standards, the specific gravity of natural soils and cement particles are measured with deionized water (ASTM D854-14) and kerosene (ASTM C188-17, *Standard Test Method for Density of Hydraulic Cement*), respectively. In this study, to avoid hydration of anhydrous cement in the soil, kerosene, instead of water, was also used when measuring the specific gravities of cement-stabilized clays. Five replicate samples for each drying method were measured for specific gravity. A vacuum pressure of 80 kPa was used to remove entrapped air in stabilized soil particle-kerosene mixture with a duration of 6 h for measuring the specific gravity. The measurement errors, defined as discrepancy percentages in water content and specific gravity of four oven-drying methods (OD110, ED110, OD60, and ED60) with respect to those of freeze-drying method (FD), were calculated as follows

$$D_w = \frac{|\bar{w} - \bar{w}_{FD}|}{\bar{w}_{FD}} \times 100 \% \quad (2)$$

$$D_{Gs} = \frac{|\overline{Gs} - \overline{Gs_{FD}}|}{\overline{Gs_{FD}}} \times 100 \% \quad (3)$$

where D_w and D_{Gs} are the discrepancy percentages in water content and specific gravity, respectively. $\overline{w}$ and $\overline{Gs}$ are the average values of water content and specific gravity, respectively. $\overline{w_{FD}}$ and $\overline{Gs_{FD}}$ are the average values of water content and specific gravity, respectively, in the freeze-drying method.

For X-ray diffraction (XRD) tests, the dry cement-clay mixture was prepared as a control. Referring to the typical particle size for XRD measurements ranging from 10–50 μm (Pecharsky and Zavalij 2009), all samples dried by five methods were further manually ground and sieved through the sieve of 38 μm (ASTM No. 400 sieve). Although water has little effect on the XRD patterns, the wet cement-stabilized clay with a particle size less than 50 μm is difficult to prepare. Therefore, the XRD test was conducted on dried samples. The XRD tests were conducted using a Bruker D8 Advance diffractometer with $\text{CuK}\alpha$ radiation set to 40 kV and 40 mA. The XRD patterns of selected specimens were analyzed using the Jade 6 software.

Results and Discussion

XRD

The XRD patterns of stabilized clay samples dried by different methods are shown in figure 1. The result of the dry clay-cement mixture with a cement content of 30 % is also provided in figure 1, labeled as “BH” (before hydration). In the dry mixture, the main minerals in the clay, including kaolinite, illite, and quartz, were observed; the typical anhydrous cement components, including alite (C_3S), belite (C_2S), and aluminate (C_3A), were also identified. For the stabilized clay, CSH, the main cement hydration product, was detected in samples dried by all five methods. However, other cement hydration products, including ettringite and hemicarboaluminate (Hc), were only observed in OD60, ED60, and FD samples. The peaks of ettringite cannot be confidently identified in the samples dried at 110°C (OD110 and ED110), which is attributed to the dehydration of ettringite starting at the temperature of 70°C–90°C (Zhou and Glasser 2001; Shimada and Young 2001). For Hc, when the temperature increases up to 75°C, it can partially transform to carbonated Hc with a decrease in the intensities in XRD patterns (Runčevski et al. 2012). Therefore, compared with OD60, ED60, and FD samples, the major peak ($2\theta = 11.1^\circ$) for Hc decreased significantly in OD110 and ED110 samples. Unlike the ettringite, peaks of CSH were observed in all samples cured for both 7 and 180 days because the dehydration temperature of CSH is higher than 110°C (Stepkowska et al. 2004; Alonso and Fernandez 2004).

At 7 days (fig. 1A), the peaks of OD60 and ED60 samples at $2\theta = 29.4^\circ$ were significantly higher than those of FD, OD110, and ED110 samples. Both CSH ($\text{Ca}_{1.5}\text{SiO}_{3.5}\cdot x\text{H}_2\text{O}$, $2\theta = 29.355^\circ$) and calcite ($2\theta = 29.405^\circ$) have peaks at $2\theta = \sim 29.4^\circ$. However, CSH does not exhibit sharp peaks in XRD diffractogram because of its amorphous nature; hence, the increase in intensities of OD60 and ED60 samples should be mainly due to the production of calcite (CaCO_3) from the carbonation of anhydrous cement (i.e., C_3S and C_2S), as shown in equations (1) and (2) (Peter et al. 2008). At 7 days, peaks at $2\theta = 29.4^\circ$ in samples dried at 110°C were lower than those of samples dried at 60°C. This should be due to the fact that the water required in the carbonation reaction was removed quickly at 110°C; therefore, the amount of produced calcite decreased. When cured for 180 days (fig. 1B), no significant difference in the peak at $2\theta = \sim 29.4^\circ$ between samples dried at 110°C and 60°C was observed, and peaks of OD60 and ED60 samples cured for 180 days were lower than those of samples cured for 7 days because very little anhydrous cement was left to be carbonated during the drying process after 180 days.

No significant intensity difference was observed between samples with and without ethanol (i.e., ED60 versus OD60; ED110 versus OD110). It is possible that the high temperature (60°C or 110°C) accelerated cement

FIG. 1 X-ray diffraction patterns of cement-stabilized soft clay with curing (A) 7 days and (B) 180 days. A = alite (C_3S); Al = aluminate (C_3A); B = belite (C_2S); BH = before hydration; C = calcite; CSH = calcium silicate hydrate; E = ettringite; Hc = hemicarboaluminate; I = illite; K = kaolinite; M = Mica; Q = quartz; S = smectite.

hydration reactions in the stabilized clay during oven-drying, but it is difficult to detect the changes by XRD because the main hydration product, CSH, is amorphous.

WATER CONTENT

Figure 2 shows the water contents of cement-stabilized clay samples dried by different methods. The initial water content of the wet cement-clay mixture before cement hydration was 69.2 %. Because the cement hydration consumed water, the water content of stabilized clay decreased with increasing curing time. As shown in **figure 2**, for a fixed curing time and oven temperature, the effect of ethanol on water content measurement of stabilized clay was limited, with the discrepancy percentage in water content measurement caused by the addition of ethanol only ranging from 0.5 to 2.3 %. This indicates that the cement hydration during the oven-drying process is limited. This is because most of the water in the stabilized clay is dried very quickly and is not available for cement hydration reactions.

As shown in **figure 2**, for the same curing time, the water contents of samples oven-dried at 110°C and 60°C were higher and lower, respectively, than those measured by freeze-drying. The higher water content measured for soil samples oven-dried at 110°C was mainly because some crystalline water was also removed from hydration products (e.g., ettringite and Hc) when oven-dried at 110°C, as indicated by the XRD results shown in **figure 1**.

FIG. 2

Water content of cement-stabilized soft clay dried by different methods.

The bound water is strongly attracted to soil mineral surfaces, especially for clays (Mitchell and Soga 2005), and a high temperature ($>60^{\circ}\text{C}$) is required to remove the bound water from the clay surfaces (Gardner et al. 2001; Logsdon and Laird 2004). The organic solvent is strongly absorbed in cement hydration products and cannot be entirely removed by the oven-drying method (Thomas 1989; Beaudoin et al. 1998). Furthermore, both boiling temperatures of water (100°C) and ethanol (78.4°C) are higher than the oven temperature of 60°C . Therefore, the oven-drying at 60°C cannot completely remove the ethanol and free and bound water from the stabilized clay.

At 7 days, the water content of OD110 was similar to that of ED110, whereas the water content of OD60 was slightly smaller than that of ED60. This is because, at the temperature of 110°C , free and bound water was removed quickly; hence, the effect of ethanol on water content measurement was limited. The free and bound water was not removed completely at the temperature of 60° , and cement hydration in OD60 led to lower water content, whereas ethanol serving for arresting hydration contributed a slightly higher water content in ED60. At 180 days, the water content of OD110 was slightly higher than that of ED110. A possible reason is that the interaction between ethanol and hydration products (Scrivener, Snellings, and Lothenbach 2016) in ED110 might lead to a lower water content. The small difference in water content between OD110 and ED110 might be attributed to the bias in measurement, considering the error bar in OD110 (fig. 2). The water content of OD60 was similar to that of ED60 because of the insignificant effect of ethanol on water content measurement in stabilized clay cured for 180 days with limited cement hydration reactions.

Table 2 summarizes the measurement errors of four oven-drying methods with respect to the freeze-drying method, referring to equations (2) and (3). For stabilized clay cured for 7 days, OD110 and ED110 had relatively lower measurement errors (3.06 and 3.63 %, respectively) compared with OD60 and ED60 (9.44 and 7.36 %, respectively). However, all four methods had similar measurement errors (4.02–5.58 %) for stabilized clay cured for 180 days. For OD110 or ED110, the measurement error increased slightly as the curing time

TABLE 2

Summary of measurement errors of oven-drying methods with respect to the freeze-drying method

Curing Time	7 days		180 days	
	D_w , %	D_{Gs} , %	D_w , %	D_{Gs} , %
OD110	3.06	0.44	5.54	0.66
ED110	3.63	0.37	4.02	1.59
OD60	9.44	0.93	5.58	0.60
ED60	7.36	0.19	5.28	2.17

increased from 7 to 180 days, which is because the amount of hydration products increases with increasing curing time and, therefore, more crystalline water can be removed in the stabilized clay cured for a long time. However, for OD60 or ED60, the measurement error decreased when the curing time increased from 7 to 180 days. The bound water in clay particles is strengthened by the large specific surface areas (Mitchell and Soga 2005). As curing time increases, more hydration products are formed to fill pores by decreasing the porosity of soil, which decreases the specific surface area and, hence, weakens the attraction force between bound water and clay particles. Therefore, it is relatively easier to remove bound water in stabilized clay cured for 180 days than that cured for 7 days.

SPECIFIC GRAVITY

Figure 3 shows the specific gravities of stabilized clay dried by different methods. The specific gravity of the dry clay-cement mixture before hydration was 2.8. After hydration, the specific gravity decreased with increasing curing time primarily because of hydration products with relatively lower specific gravity, e.g., specific gravity of ettringite = 1.77, specific gravity of CSH = 2.2–2.5 (Jennings 2000), by comparing with the high specific gravity of 3.15 for anhydrous cement and 2.65 for dry clay. As shown in **Table 2**, compared with water content measurement, the effect of drying method on the specific gravity of stabilized clay was insignificant, with a measurement error ranging from 0.19 to 2.17 %.

As shown in **figure 3**, at 7 days, the specific gravity of OD60 was higher than other drying methods. This is because, compared with samples dried at 110°C (OD110 and ED110), the oven temperature of 60°C on hydration was less significant and fewer hydration products (e.g., CSH and ettringite) with low specific gravities were formed, which contributes a high specific gravity in OD60. Compared with FD, a slightly higher specific gravity of OD60 was due to the formed calcite (specific gravity = 2.71) as discussed previously.

At 180 days, the specific gravity measured by four oven-drying methods was higher than that of FD. As shown in **Table 2**, the two maximum measurement errors were obtained for the samples dried with ethanol at 180 days, i.e., ED110 (1.59 %) and ED60 (2.17 %), with respect to FD, which may be attributed to the physical interactions between ethanol and hydration products in the stabilized clay. The physical interactions are due to the similar polarity and the availability of space for chemical bonds in the hydration products between organic fluids and hydration products (Scrivener, Snellings, and Lothenbach 2016), resulting in ethanol trapped and a slight increase in specific gravity of the stabilized clay. However, the trend of higher specific gravity in samples dried with ethanol is not observed in samples cured for 7 days. This is because the effect of interactions between hydration products (e.g., CSH) and ethanol on the specific gravity of stabilized samples is not significant because of the small amount of hydration products at 7 days. The specific gravity of OD110 or OD60 was also higher than that of FD because some crystalline water was removed during the oven-drying process.

FIG. 3

Specific gravity of cement-stabilized soft clay dried by different methods.

Conclusions

The following conclusions are drawn from this study:

1. XRD results of cement-stabilized clay samples dried by different methods indicated that the high oven temperature of 110°C could change some cement hydration products as evidenced by the peaks recorded by XRDs of the treated soils.
2. Water content measured by oven drying at 110°C was higher than that measured by the freeze-drying method because some hydration water was removed, whereas the water content measured by oven drying at 60°C was lower than that measured by the freeze-drying method because the bound water was not completely removed. For 7-day cement-stabilized clay, oven drying at 110°C achieved lower errors (3.06–3.63 %) than oven drying at 60°C (7.36–9.44 %) in water content measurement. For 180-day cement-stabilized clay, all the oven-drying methods had similar measurement errors (4.02–5.58 %). The effect of ethanol on the water content measurement of stabilized clay was insignificant.
3. The effect of the drying method on the specific gravity measurement was insignificant, with the percentage of measurement error ranging from 0.19 to 2.17 %.
4. In summary, compared with conventional oven drying at 110°C, there was no advantage to using ethanol and a low temperature of 60°C for the measurement of water content and specific gravity of cement-stabilized clay.

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