Geomechanics and Engineering, *Vol. 27, No. 6* (*2021*) *573-582*
https://doi.org/10.12989/gae.2021.27.6.573

Effect of bound water on mechanical properties of typical subgrade soils in southern China

Le Ding[1a], Junhui Zhang[*1] and Zonghuang Deng[2b]

[1]*National Engineering Laboratory of Highway Maintenance Technology, Changsha University of Science & Technology, Section 2, No.960, Wanjiali South Road, Changsha, China*
[2]*Jiangxi Tianchi Expressway High-Tech Development Co., Ltd., Jiangxi Provincial Expressway Investment Group, Co., Ltd, No.367, Chaoyangzhou Mid-road, Nanchang, China*

(*Received May 20, 2020, Revised November 8, 2021, Accepted December 1, 2021*)

Abstract. From the effect of bound water, this study aims to seek the potential reasons for difference of mechanical experiment results of subgrades soils. To attain the comparatively test condition of bound water, dry forming (DF) and wet forming (WF) were used in the specimen forming process before testing, series of laboratory tests, i.e., CBR tests, direct shear tests and compaction tests. The measured optimal moisture contents, maximum dry densities, CBR, cohesion *c,* and internal friction angle *φ* were given contrastive analysis. Then to detect the adsorptive bound water in the subgrade soils, the thermal gravimetric and differential scanning calorimetry (TG-DSC) test were employed under different heating rates. The free water, loosely bound water and tightly bound water in soils were qualitatively and quantitatively analyzed. It was found that due to the different dehydration mechanics, the lost bound water in DF and WF process show their own characteristics. This may lead to the different mechanical properties of tested soils. The clayey particles have a great influence on the bound water adsorbed ability of subgrade soils. The more the clay content, the greater the difference of mechanical properties tested between the two forming methods. Moreover, in highway construction of southern China, the wet forming method is recommended for its higher authenticity in simulating the subgrade filed humidity.

Keywords: bound water; clayey content; differential scanning calorimetry; specimen forming; subgrade soil

1. Introduction

Bound water in soil is the product of long-term interaction between mineral particles and vapor or aqueous solution under circumstances (Caurie 2011, Wang *et al.* 2020). Since it is mainly attached to the surface of clayey mineral particles, bound water shows different states under diverse environmental and force conditions. Many relevant experimental researches on the type and property of soils' internal bound water had been carried out. According to the electrostatic attraction of water molecules, existing forms and activities, the distance from the surface of mineral particles, bound water is classified as two categories: loosely bound water and tightly bound water(Low 1979, Ma and Hueckel 1992, Zymnis *et al.* 2019). Some laboratory tests showed that the thickness and surface area of water molecules on soil particles was associated with the viscosity of the clay-water system, the conductivity of clay solution, contact mode between soil particles(Singh and Wallender 2008, Xu *et al.* 2019). The influence of cation exchange on the bound water were also studied in expansive soil(Mojid and Cho 2012, Tang and Cui 2009). It is generally believed that, the complexity and variety of bound water is found to be effective in the soil basic properties significantly, such as hydrophilicity, dilatancy, plasticity, and mechanical strength(Kleinberg and Griffin 2005, Panchev *et al*. 2005). For detection methods, some physical, chemical, geological tests were mainly used to classify and analyze bound water in soils recently years(Kasprzhitskii *et al*. 2016, Logsdon and Laird 2004, Watanabe *et al*. 2002). For instance, isothermal adsorption test, thermo-gravimetric analysis, and differential scanning calorimetry test (TG-DSC) were proved to be capable of detecting the content of bound water on the soil surfaces(Souza and Nascimento 2008, Tomura *et al*. 1998, Wang *et al*. 2011b).

As the carrier of the bound water, the clayey mineral is a kind of tiny flat and fibrous particle, belongs to the fine-grained group according to China's standard *Test Methods of Soils for Highway Engineering*(JTG-E40-2007). From microcosmic investigation, the surface of the mineral particle is usually strongly interacted with the water molecules. The smaller the particle size, the larger the specific surface area with stronger absorbed ability(Yeşilbaş and Boily 2016). Different kinds of soils show their own particular properties ascribed from the effect of bound water(Hilhorst *et al*. 2001, Sposito and Prost 1982).

In highway engineering, an excessive highly moisture content is proved to be a critical factor causing a series diseases of pavement and subgrades(Morrow *et al*. 2000, Tian *et al.* 2014), but little work has been done in the effect

*Corresponding author, Professor
E-mail: zjhseu@csust.edu.cn
[a]Ph.D.
E-mail: Leding@stu.csust.edu.cn
[b]Ph.D.
E-mail: dengzh93@163.com

Copyright © 2021 Techno-Press, Ltd.
http://www.techno-press.org/?journal=gae&subpage=7
ISSN: 2005-307X (Print), 2092-6219 (Online)

of bound water on mechanical properties of subgrade soils. Simultaneously, due to the hot and humid climate in south China, the moisture exchange frequently between the subgrade soils and atmosphere, some subgrades remain in a long-term wet state with large amounts of bound water attached in the subgrade soils, resulting in the subgrade performances deteriorate(Nagrale and Patil 2017). Therefore, revealing the affect law of bound water on engineering properties of subgrade soils is meaningful to design and construction of durable subgrades. However, the existence of bound water in soil still does not get much attention in subgrade of highway engineering.

To address this issue, four typical subgrade soils were selected in southern China, and a series of laboratory experiments were carried out to analyze their mechanical properties in wet and dry forming methods, including CBR tests, shear tests, and compaction tests. The CBR value, cohesion and friction angle, maximum dry density and optimum moisture content were analyzed. Furthermore, the TG-DSC was used to record the reduced value of moisture content within the soil samples in different temperature and heating rates, and the corresponding temperature ranges of thermos weight loss were determined. By comparing and analyzing the clayey particle content of different soil samples, the effect of the bound water on mechanical properties of subgrade soils was revealed.

2. Materials and methods

The selected soil samples were taken from Hunan and Guizhou Province of southern China, classified as four categories based on the standard (JTG E40-2007): low-liquid-limit-sandy-clay (CLS), high-liquid-limit-silt (MH), high-liquid-limit-clay(CH), high-liquid-limit-sandy-silt (MHS). After the specific gravity test, moisture content test, clayey particle test, liquid-plastic limit combined test and the screening test were conducted, their primary physical properties can be obtained in Table. 1. It shows that the natural moisture content and clayey particle content differed greatly in each soil. The grading curves shows the content of the fine particle (d<0.075mm) in all the soils exceeds 50% (see Fig. 1), and even larger than 95% in CH and MH. Thus, they all belong to fine-grained soil according the precious standard (JTG E40-2007).

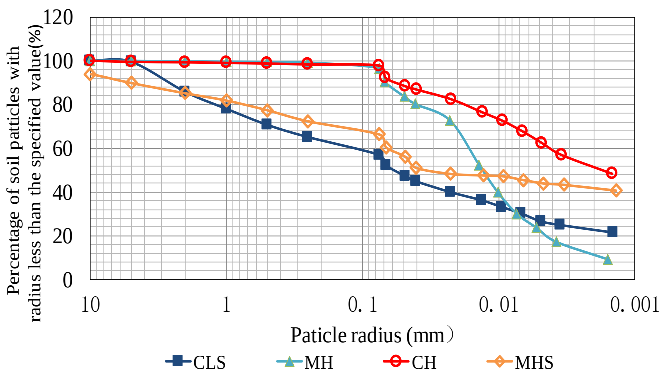


Fig. 1 Grading curve of the test soil

Table 1 Basic physical properties of typical subgrade soils

Soil type	Natural moisture content (%)	Specific gravity	Liquid limit (%)	Plastic limit (%)	Clayey particles content (d<0.002 mm) (%)
CLS	18.6	2.68	46.7	23.6	22.47
MH	26.2	2.69	51.6	31.8	9.26
CH	29.8	2.71	57.7	28.4	51.56
MHS	37.4	2.73	67.2	41.4	41.42

2.1 Mechanical performance tests

To study the effect of bound water on properties of tested soils, several groups of comparative tests, including compaction tests, CBR tests and direct shear tests, see Figs. 2(a) and 2(b), were employed in two forming methods: dry forming and wet forming, specified in China's standard (JTG E40-2007) shown in Figs. 2(c) and 2(d). In the first case, an oven was used to heat the soil at the temperature of 105ºC-110ºC, the microstructure of loosely bound water destroyed in this stage, some water molecules got lost, and the cementitious action between the soil particles couldn't restore, resulting in the irreversible destruction of the soil structure during the test. For the second: the soil samples were air-dried under the room temperature, it had no obvious damage to soil structures and more conformed to the actual condition. The mechanical property indexes of two forming conditions were compared comprehensively.

2.2 Bound water tests

For the bound water at different distance from the surface of mineral particles, their dehydrated temperatures are not the same, as well as the bound energy(Lagaly and Ziesmer 2003, Tone *et al*. 1993). Thus, it is necessary to identify the dehydrated critical temperature of free water, loosely bound water and tightly bound water. The TG-DSC method has been used extensively in soil science to define the exact temperature when dehydration events occur. It can record sample weight loss in the heating process and build a function of scanning temperature, and the DSC curve displays an endothermic or exothermic peak(Costa *et al.* 2004, Wang *et al*. 2011a). In this test, the previous soil samples (CLS, MH, CH, MHS) were used to be performed to study the content of bound water in each soil. By analyzing the location, shape, and size of heat-absorbing valley on DSC curves, the type, content and boundary temperature of the different water within soils can be determined (Hoeg *et al.* 2004), and then the mechanical strength characteristics of soils in dry forming and wet forming method were revealed.

The detection instrument used in this TG-DSC test was SDT Q600 (TA Instruments Company. USA) showed in Fig. 3. Its sensitivity is 0.001ºC with thermo-balance sensitivity of 0.1ug, and the heating rate can be adjusted from 0.1 to 50ºC/min. To eliminate the influence from sample's purity and particle size, the tested soils were screened by a 0.075mm sieve in the natural air-drying state,

(a) Cooker heating

(b) Naturally air drying

(c) Compaction test

(d) CBR test

(e) Direct shear test

Fig. 2 Specimen forming and mechanical property tests

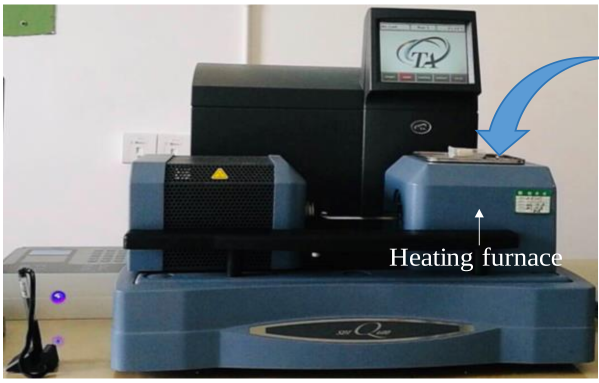


Fig. 3. Differential scanning calorimeter

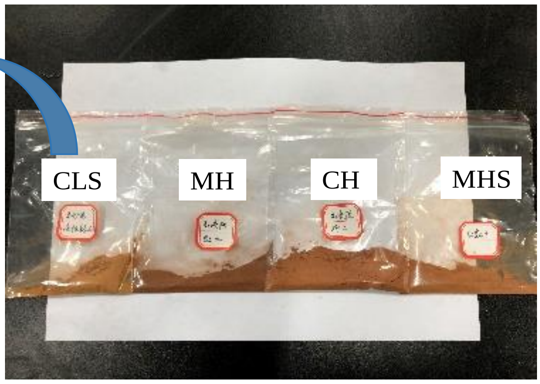


Fig. 4. Four test soil samples

see Fig. 4. Particularly, they should be tested in time to avoid absorbing moisture from the atmosphere. To ensure the accuracy and reliability of the test results, the experimental data were compared and analyzed in three heating rates:1ºC/min, 3ºC/min, and 5ºC/min. The whole test can be ended when the quality of tested samples remained constant.

3. Tests results

3.1 CBR and compaction tests

CBR curves and compaction curves of four soils are presented in Fig. 5 in two forming methods. The ω corresponded to the peak location of dry densities (ρ_{dmax}) is the optimal moisture content(ω_{omc}) that often used to guide the site construction. As the moisture content(ω) increases, the measured CBR and dry density increases initially and then decreases, and a peak exists in each curve in all the cases. But the peak location of the CBR curve is not coincide with that in the compaction curve. Therefore, in the condition of the maximum CBR, the measured moisture content (ω_{CRB}) is larger than the ω_{omc}. The difference values between these two moisture contents vary from 1.1% to 3.2% in DF, and 1.6% to 4.4% in WF.

Some studies showed that property experiment results for one soil in the condition of two forming methods differed. The possible reason is the changing of the bound water in soils caused by the forming methods(Zhang *et al*. 2020b). When ω is in a lower range, the CBR values of DF samples are larger than that of WF samples. However, with the increase of ω, the difference value of CBR between DF and WF gradually weaken, and the two CBR curves tend to be overlapped in the final stage. This can be attributed to the porosity of the specimen gradually decreases in the CBR test, and approaches to saturation state before immersing into water. The moisture content corresponded to the maximum CBR(ω_{CRB}) in DF is smaller than that in WF, but the value of maximum CBR is larger in DF, similar to the compaction test.

3.2 Direct shear tests

In the plotting of direct shear test results of CLS in Fig. 6, the normal stress P was applied as the abscissa, and the

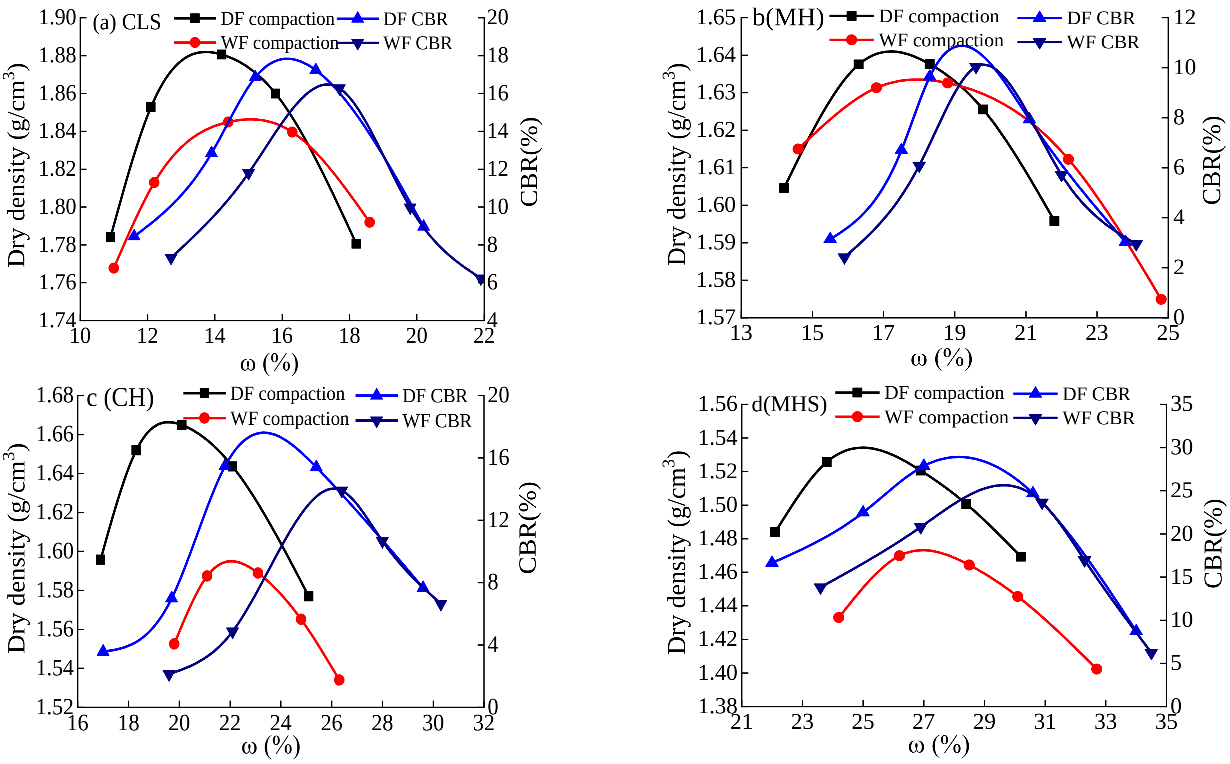


Fig. 5 CBR tests of four kinds of soils: (a)CLS, (b)MH, (c)CH, (D)MHS

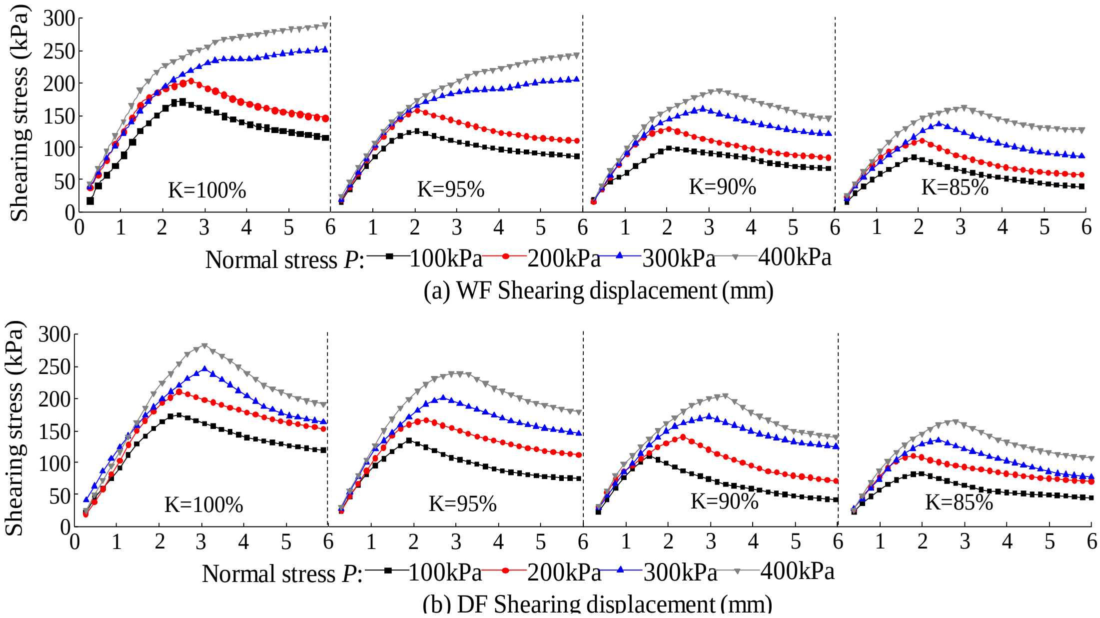


Fig. 6 Shear stress-shear displacement curve of CLS

shear strength τf in each grade stress is set as the ordinate. There are obvious peaks in the condition of DF, which shows a strain softening characteristic. For WF, most of test results also presented a same trend with a peak exist, except for four cases showed a strain hardening (K=95%, K=100% and P=300kPa, P=400kPa). This can be ascribed to the soil structure of CLS: the clayey particle content is only 22.47%, there are a bit of adsorbed bound water on the soil's surface, leading to the weak cementation between particles. However, due to its relatively high coarse grain content and large maximum dry density, CLS still has a strong cohesion and highly shear strength under the condition of optimal moisture content. Direct shear test indexes are analyzed by Molar-Coulomb Strength Theory. All the data are positioned in Fig. 7. It shows a good linear correlation between the shearing stress and normal stress. The intercepts of the line obtained by fitting analysis are cohesion forces, and the corresponding inclination angles are internal friction angles.

Fig. 8 presents the comparative analysis of shear strength indexes of four soils in different compaction degree and forming methods. It is investigated that with the increase of compaction degree(K), the cohesion(c) and internal friction angle(φ) gradually rises. The higher the compaction degree, the smaller pores and the closer contact connection between soil particles, and the higher the cohesion. The relative sliding of soils particles only occurs under the stronger shear stresses. With the increase of compaction degree, the growth rate of cohesion rises up, but it slows down for the internal friction angle. In the case of CLS, as the compaction degree varies from 85% to 100%, the cohesions increase by 78.7kPa in DF and 79.7kPa in WF, and the internal friction angles increase by 4.7° in DF and 4.1° in WF. In the same compaction degree, the internal friction angle obtained from DF is greater than that from WF, but the situation is completely converse for the cohesion. The difference of indexes between DF and WF become more obviously as the compaction degree increases. For instance, in the test of CH, when the compaction degree varies from 85%, 90% and 95% to 100%, the difference of cohesion between DF and WF is 0.423kPa, 0.538kPa, 1.820kPa and 2.613kPa, and 0.612°, 0.773°, 1.606°, and 2.04° in the internal friction angle.

3.3 Quantitative analysis of bound water tests

With the increase of the temperature in the bound water testing, three types of water in soils (free water, loosely bound water, tightly bound water) dehydrated successively accompanied by endothermic reactions. As the surface temperature of the soil particles decreased, the heat energy compensated immediately to keep the temperature balance

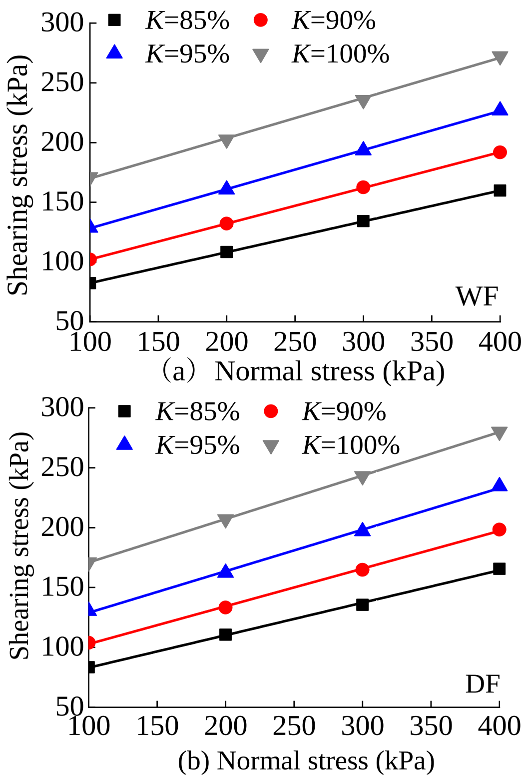


Fig. 7 Shearing strength curves of CLS

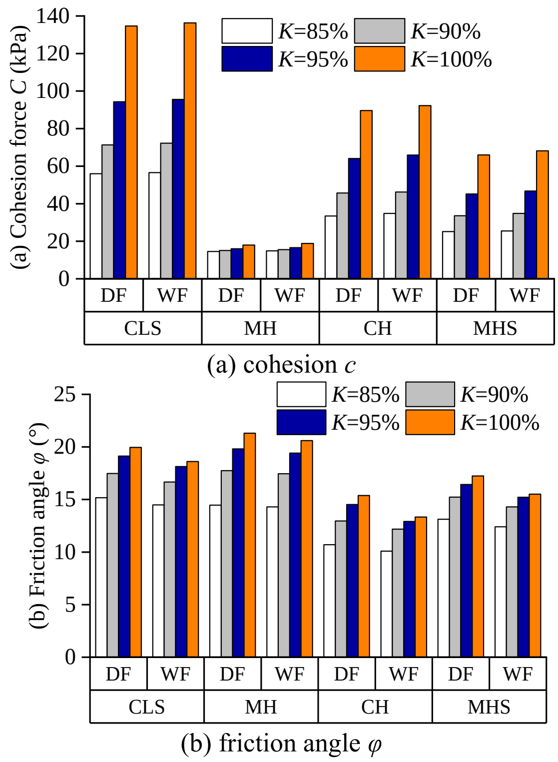


Fig. 8 Comparison of shear strength indexes

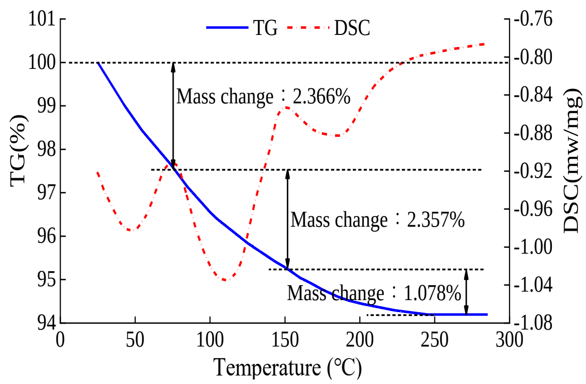


Fig. 9 TG-DSC curve of CLS at the heating rate of 1ºC/min

with reference materials. Each type of water has a critical dehydration temperature in the peak location of the DSC curve, and the required compensatory heat get maximum at this moment. Therefore, three noticeable endothermic troughs formed, the dehydration temperature range of three kinds of water in soil were revealed: the one corresponded to the loosely bound water is in the lowest position, then the free water, the tightly bound water the higher. In the process of dehydration, the loosely bound water required maximum compensatory heat, then the free water, and minimum for the tightly bound water. It also can be estimated that the dehydration temperature of tightly bound water is much higher than that of free water and loosely bound water.

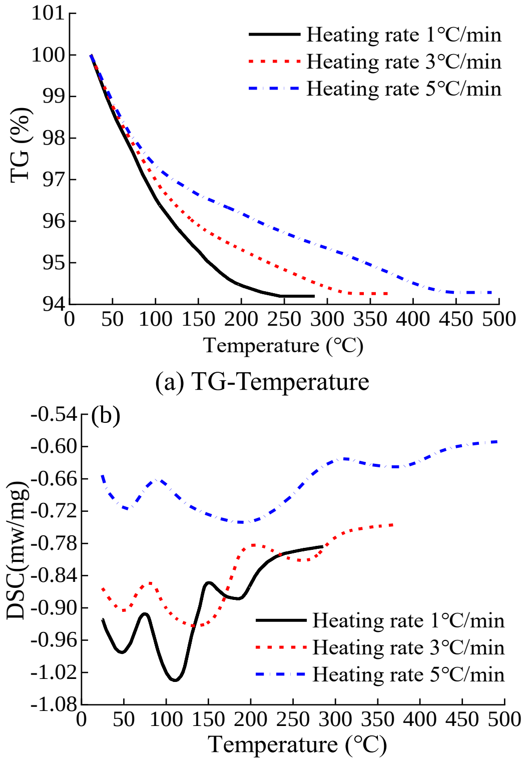
Subsequently, the contents of three types of water in soil can be acquired accurately and quantitatively by the TG curve. Fig. 9 shows the test result of CLS in the heating rate of 1ºC/min, the content of free water, loosely bound water and tightly bound water is 2.366%, 2.357%, and 1.078% respectively. Under different heating rates, the measured contents of each water and critical temperatures also differed, depicted in Fig. 10. As the heating rate increases, it is 2.405%, 2.315%, 1.022% in 3ºC/min respectively, and 2.438%, 2.286%, 0.991% in 5ºC/min. With the increase of the temperature, the position of the loosely bound water and the tightly bound water in the heat-absorbing valleys both show an upward and right-shift trend, and the area and peak's width of heat-absorbing valley of loosely bound water get larger significantly, indicated the dehydration temperature range may be broadened for the bound energy of the loosely bound water is weak(Siewert and Kučerík 2015). By the same method, the TG-DSC curves of other three soils were detected, the precise critical temperatures and the contents of absorbed water can be calculated out, see Table 2.

4. Discussion

4.1 Comparison of the compaction indexes of DF and WF

From the indexes (ω_{omc} and ρ_{dmax}) obtained from compaction tests in the condition of DF and WF, the effects of bound water can be analyzed qualitatively. In the DF compaction test, as the temperature in the oven is usually set at 105ºC-110ºC, the free water and most of loosely bound water were taken off, but it had no effect on the tightly bound water. The fine particles with gel properties inside the soil (Fe2O3•nH2O, SiO2•nH2O) gradually dehydrated and solidified, and the films on the surface of soil particles became thinner, making the soil particles contract and condense continuously(Tuller and Or 2005), the internal porosities decreased significantly. Therefore, the maximum dry density obtained in DF is larger due to the more compacted soil become in this condition.

For the compaction test of WF, the soil particles were fully condensed in a dynamic drainage consolidation stage(Tang *et al.* 2018). With the number of compaction cycles gradually increased, the film of bound water was continuously compressed, the electrostatic attraction on the clayey particles got weak until completely destroyed. Then some part of loosely and tightly bound water were converted into free water and attached on the surface of soil particles. In DF compaction, the soils internal structure got destroyed, making the condensation and contraction between soil particles non-uniformed, and difficult for the bound water to be converted into free water. Thus, the measured moisture content in WF is larger than that in DF. It is revealed that in some previous studies, the moisture content of subgrade soils increase gradually and eventually reach a humidity balance state, close to the plastic limit ω_p of the subgrade soils(Peng *et al*. 2020, Teltayev and Suppes 2017). For instance, in CLS, six kinds of moisture contents, ω_{omc} and ω_{CRB} in DF and WF, and the equilibrium moisture content ω_e and plastic limit ω_p are compared in Fig. 11. The ω_{omc} in DF is 13.5%, smaller than WF. And the ω_{CRB} obtained in WF is 17.3%, closer to ω_p 23.6% and ω_e 24%, the moisture state of CBR specimens in WF method is more similar to that of the construction in-site.



(b) DSC-Temperature

Fig. 10 Comparison of TG-DSC curves of CLS samples at different heating rates

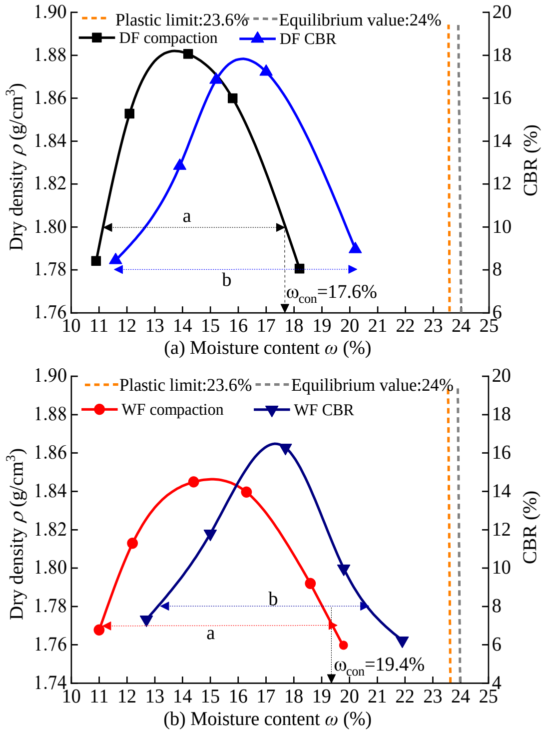


Fig. 11 Determination of moisture content of CLS in the field construction

Therefore, in the qualified ranges of compaction degree and CBR, a determination method of construction moisture content ω_{con} can be proposed. For instance, in China's standard of subgrade design (JTGD 30-2015), in the upper layer of subgrade within the thickness of 30 cm, the compaction degree should be greater than 96%, it falls in the range (a) in Fig. 10. Meanwhile, the CBR value should be greater than 8% in the range (b). Thus, the ω_{con} should be taken in the overlap part of two ranges. Simultaneously, to keep the durability of highway in the later operation stage, it is supposed to be close to ωe. The ω_{con} obtained from WF is closer to ω_e(17.6% in DF and 19.4% in WF). Thus, the WF method was recommended in determining the designed moisture content in the construction, since it is more conducive to improve the subgrade's stability and durability(Zhang *et al.* 2020a).

4.2 Bound water content and its temperature characteristic

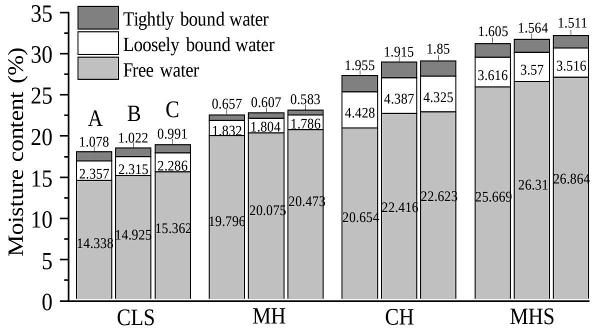
According to the TG-DSC curve, the percentage of loosely bound water and tightly bound water in soil samples can be measured quantitatively. The lost water in DF(*n*) include all the free water and most of the loosely bound water. The relationship between natural moisture content *ω* and the percentage of lost water within soils in DF(*n*) is revealed in Eq. (1) The difference value between lost water in DF and lost loosely bound water is the free water, see Eq. (2) Simultaneously, the percentage of loosely bound water dehydrated from the initial temperature to 105ºC was obtained from the TG-DSC curve.

$$n = \frac{\omega}{1+\omega} \tag{1}$$

$$\omega_f = n - \omega_l \tag{2}$$

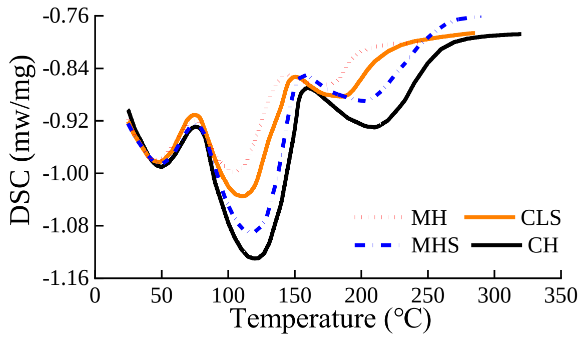
Where ω-natural moisture content(%); n-percentage of lost waste in DF(%); ω_f-percentage of free water(%); ω_l-percentage of lost loosely bound water(%).

The measured three types of water contents in four soils under different heating rate are shown in Fig. 12. As the heating rate exceeds one certain value, the free water content measured by DSC increase, but the content of loosely bound water and tightly bound water decrease. In the case of loosely bound water, even if it reaches to the critical temperature, free water covered on its surface doesn't completely decrease due to the rapid heating rate, some part of loosely bound water gets dehydrated only in a higher temperature range. It is implied that with the increase of heating rates, the critical dehydrate temperature of each type of water also increases, the corresponding positions of heat-absorbing valleys show a backward trend. Parts of loosely bound water shows the characteristic of free water, and parts of tightly bound water shows the characteristic of loosely bound water, making their measured contents smaller than their actual contents. Therefore, the heating rate of 1ºC/min is recommended to be selected in the test to make the measured content accurately.

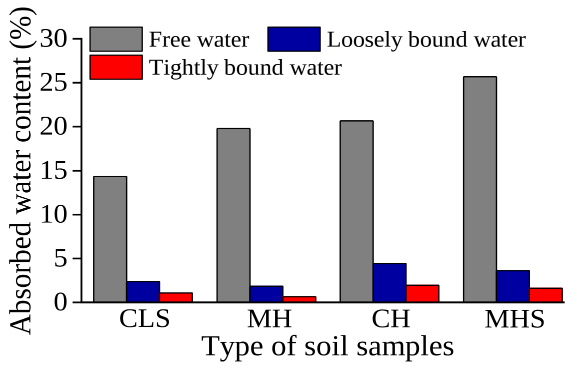


*Note: heating rate: A, 1ºC/min; B, 3ºC/min; C, 5ºC/min

Fig. 12 Comparison of adsorbed water measured at different heating rates



(a) DSC curve



(b) Absorbed water content

Fig. 13 Comparison of bound water tests of four kinds of soils at the heating rate of 1ºC/min

Fig. 13(a) shows the DSC curves of the four soils under the heating rate of 1ºC/min, and the comparison of the measured adsorbed water contents, presented by Fig. 13(b). The size and location of heat-absorb valleys corresponded to free water are identical, but significantly differ for loosely bound water and tightly bound water. Since the free water is usually free from electrostatic attraction, the hydrostatic pressures can be transferred for its dissolving capacity, thus the properties of free water are not affected by soil structure, but closely related to chemical mineral composition, seasonal climate, and local hydrogeological conditions. Although The TG-DSC tests of different soils show the same characteristics and variation trend, three types of water show its own thermal weight loss range, 25ºC-75ºC for free water, 75ºC-160ºC and 143ºC-276ºC in loosely and tightly bound water respectively.

Table 2 Comparison of measured water contents in different heating rates

Heating Rate (℃/min)		Free water IT (℃)	Free water CDT (℃)	Free water Content (%)	Loosely bound water IT (℃)	Loosely bound water CDT (℃)	Loosely bound water Content (%)	Tightly bound water IT (℃)	Tightly bound water CDT (℃)	Tightly bound water Content (%)
CLS	1	25	74	2.366	74	150	2.357	150	245	1.078
	3	25	81	2.405	81	204	2.315	204	329	1.022
	5	25	90	2.438	90	310	2.286	310	446	0.991
MH	1	25	74	2.676	74	144	1.832	144	226	0.657
	3	25	81	2.694	81	182	1.804	182	278	0.607
	5	25	88	2.734	88	268	1.786	268	376	0.583
CH	1	25	75	2.512	75	160	4.428	160	276	1.955
	3	25	86	2.535	86	231	4.387	231	376	1.915
	5	25	93	2.598	93	406	4.325	406	558	1.850
MHS	1	25	74	5.217	74	157	3.616	157	268	1.605
	3	25	83	5.253	83	220	3.570	220	360	1.564
	5	25	93	5.284	93	366	3.516	366	512	1.511

Note: IT=Initial dehydrated temperature; CDT=Critical dehydrated temperatu

Table 3 Comparison of characteristics of typical subgrade soils

Soil type	Clayey particle content (%)	Specific gravity	Loosely bound water content (%)	Tightly bound water content (%)	WF compaction test ω_{omc} (%)	WF compaction test ρ_{dmax} (g/cm³)	DF compaction test ω_{omc} (%	DF compaction test ρ_{dmax} (g/cm³)
CLS	22.47	2.68	2.357	1.078	13.9	1.881	15.0	1.846
MH	9.26	2.69	1.832	0.657	17.5	1.640	18.0	1.633
CH	51.56	2.71	4.428	1.955	19.7	1.665	22.1	1.593
MHS	41.42	2.73	3.616	1.605	25.0	1.531	26.9	1.472

4.3 Analysis of the effect of clayey particles on the bound water

When the clayey particles are contacted with water, the water molecules near the soil particles are adsorbed on their surfaces under the electrostatic attraction of atoms and ions. The clayey particle content is a main factor that determine the amount of loosely bound water and tightly bound water(Zeng *et al.* 2020). To analyze the relation between the clayey particles content and bound water content qualitatively, the tested data of four soils under WF and DF are listed in Table 3.

Three defined indexes (ω_s-bound water content, $\Delta\omega$-different ω_{omc} between the case DF and WF, $\Delta\rho/s$-ratio of difference value of ρ_{dmax} between DF and WF and specific gravity) were correlated with clayey particle contents in the power exponent model, shown Fig. 14. The determination coefficients of R^2 all exceed 0.95, indicated that the (ω_s) can be calculated out precisely by the clayey particle content, as the same as $\Delta\omega$ and $\Delta\rho/s$. While the ω_{omc} is regarded as the macroscopic representative index, the reason for the deviation between two ω_{omc} obtained from the two forming methods come from the difference in the clayey particle content. The more clayey content, the more water content of the surface adsorption, greater differences exist in the ω_{omc} and ρ_{dmax} between DF and WF.

5. Conclusions

To reveal the effect of bound water on mechanical properties of typical subgrade soils in southern China, some laboratory tests, i.e., compaction tests, CBR tests and direct shear tests were carried out in two specimen forming methods: dry forming and wet forming. The bound water content of different soils was quantitatively analyzed by TG-DSC testing methods, the following conclusions can be summarized:

- For all the tested soils, the more clayey particle content, the greater difference exists in maximum dry density and optimum moisture content obtained from the compaction test of two forming methods. The same law was shown in the cohesion and internal friction angles obtained in the direct shear tests.
- As the effect of the heating rate cannot be neglected in TG-DCS tests, it should not exceed 1ºC/min to ensure the measured accuracy. The less clayey content, the less content of adsorbed bound water, the less bound energy and lower dehydration temperature of the adsorbed water.
- Since the humidity of WF samples is more similar to the state of the field subgrade soils, it is suggested that the mechanical property indexes obtained by WF method are more conducive in guiding the

construction, especially for high-liquid limited soil with highly natural moisture content and more clayey content.

Acknowledgments

The authors gratefully acknowledge the financial support offered by the National Science Fund for Distinguished Young Scholars (52025085), National Natural Science Foundation of China (51878078), Training Program for High-level Technical Personnel in Transportation Industry (2018-025). The Science and Technology Innovation Program of Hunan Province (2020RC4048). Key Research Program of Hunan Province(2022SK2083).

References

Caurie, M. (2011), "Bound water: its definition, estimation and characteristics", *Int. J. Food Sci. Technol.* **46**(5), 930-934, http://doi.org/10.1111/j.1365-2621.2011.02581.x.

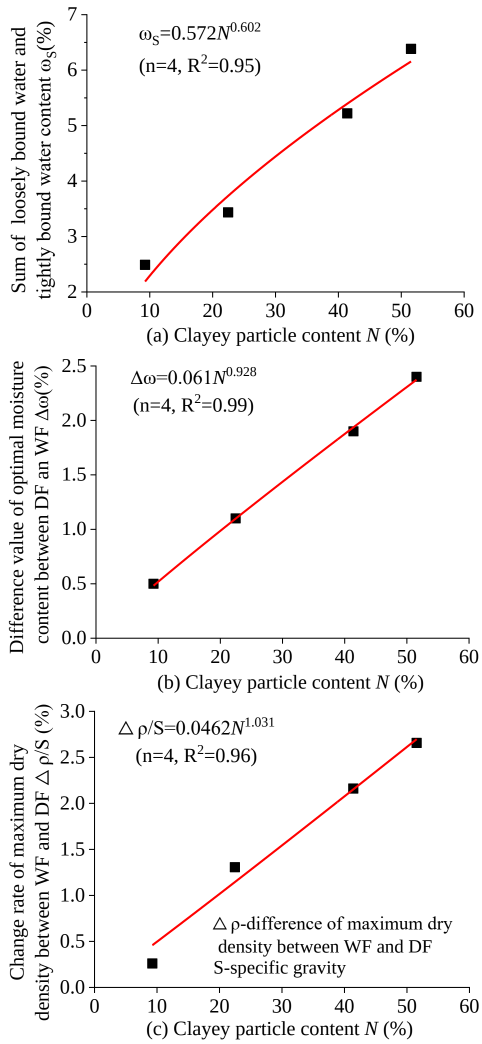


Fig. 14 Correlation of soils properties and its clayey particles content

Costa, A.C.S., Bigham, J.M., Tormena, C.A., Pintro, J.C. (2004), "Clay mineralogy and cation exchange capacity of Brazilian soils from water contents determined by thermal analysis", *Thermochim. Acta*, **413**(1), 73-79. http://doi.org/10.1016/j.tca.2003.10.009.

Hilhorst, M.A., Dirksen, C., Kampers, F.W.H. and Feddes, R.A. (2001), "Dielectric Relaxation of Bound Water versus Soil Matric Pressure", *Soil Sci. Soc. Am. J.*, **65**(2), 311-314. http://doi.org/10.2136/sssaj2001.652311x.

Hoeg, S., Schöler, H.F. and Warnatz, J. (2004), "Assessment of interfacial mass transfer in water-unsaturated soils during vapor extraction", *J. Contam. Hydrol.*, **74**(1-4), 163-195. http://doi.org/10.1016/j.jconhyd.2004.02.010.

Kasprzhitskii, A.S., Lazorenko, G.I., Sulavko, S.N., Yavna, V.A. and Kochur, A.G. (2016), "A study of the structural and spectral characteristics of free and bound water in kaolinite", *Opt. Spectrosc.* **121**, 357-363. http://doi.org/10.1134/S0030400X16090113.

Kleinberg, R. and Griffin, D. (2005), "NMR measurements of permafrost: unfrozen water assay, pore-scale distribution of ice, and hydraulic permeability of sediments", *Cold Reg. Sci. Technol.*, **42**(1), 63-77. http://doi.org/10.1016/j.coldregions.2004.12.002.

Lagaly, G. and Ziesmer, S. (2003), "Colloid chemistry of clay minerals: the coagulation of montmorillonite dispersions", *Adv. Colloid Interface Sci.*, **100**, 105-128. http://doi.org/10.1016/S0001-8686(02)00064-7.

Logsdon, S.D. and Laird, D.A. (2004), "Electrical conductivity spectra of smectites as influenced by saturating cation and humidity", *Clays Clay Miner.*, **52**(4), 411-420. http://doi.org/10.1346/ccmn.2004.0520402.

Low, P.F. (1979), "Nature and properties of water in montmorillonite-water systems", *Soil Sci. Soc. Am. J.*, **43**(4), 651-658. http://doi.org/10.2136/sssaj1979.03615995004300040005x.

Ma, C. and Hueckel, T. (1992), "Stress and pore pressure in saturated clay subjected to heat from radioactive waste: a numerical simulation", *Can. Geotech. J.*, **29**(6), 1087-1094. http://doi.org/10.1139/t92-125.

Mojid, M. and Cho, H. (2012), "Effects of water content and temperature on the surface conductivity of bentonite clay", *Soil. Res.*, **50**(1), 44-49. http://doi.org/10.1071/SR11228.

Morrow, C., Moore, D.E. and Lockner, D. (2000), "The effect of mineral bond strength and adsorbed water on fault gouge frictional strength", *Geophys. Res. Lett.*, **27**(6), 815-818, http://doi.org/10.1029/1999GL008401.

Nagrale, P.P. and Patil, A.P. (2017), "Improvement in engineering properties of subgrade soil due to stabilization and its effect on pavement response", *Geomech. Eng.*, **12**(2), 257-267, http://doi.org/10.12989/gae.2017.12.2.257.

Panchev, I., Baeva, M. and Lambov, S. (2005), "Influence of edible films upon the moisture loss and microstructure of dietetic sucrose-free sponge cakes during storage", *Drying Technol.*, **23**(4), 925-940. http://doi.org/10.1081/DRT-200054241.

Peng, J., Zhang, J., Li, J., Yao, Y. and Zhang, A. (2020), "Modeling humidity and stress-dependent subgrade soils in flexible pavements", *Comput. Geotech.*, **120**, 103413. http://doi.org/10.1016/j.compgeo.2019.103413.

Siewert, C. and Kučerík, J. (2015), "Practical applications of thermogravimetry in soil science", *J. Therm. Anal. Calorim.* **120**(1), 471-480. http://doi.org/10.1007/s10973-014-4256-7.

Singh, P.N. and Wallender, W.W. (2008), "Effects of adsorbed water layer in predicting saturated hydraulic conductivity for clays with Kozeny–Carman equation", *J. Geotech. Geoenviron. Eng.*, **134**(6), 829-836, http://doi.org/10.1061/(ASCE)1090-0241(2008)134:6(829).

Souza, C. and Nascimento, R. (2008), "Adsorption behavior of cationic polymers on bentonite", *J. Therm. Anal. Calorim.*, **94**(2), 579-583. http://doi.org/10.1007/s10973-007-8774-4.

Sposito, G. and Prost, R. (1982), "Structure of water adsorbed on smectites", *Chem. Rev.*, **82**(6), 553-573, http://doi.org/10.1021/cr00052a001.

Tang, A. and Cui, Y. (2009), "Modelling the thermo-mechanical volume change behaviour of compacted expansive clays", *Geotechnique*, **59**(3), 185-195. http://doi.org/10.1680/geot.2009.59.3.185.

Tang, L.S., Chen, H.K., Sun, Y.L., Zhang, Q.H. and Liao, H.R. (2018), "Traffic-load-induced dynamic stress accumulation in subgrade and subsoil using small scale model tests", *Geomech. Eng.*, **16**(2), 113-124. http://doi.org/10.12989/gae.2018.16.2.113.

Teltayev, B.B. and Suppes, E.A. (2017), "Regularities for temperature variation in subgrade of highway", *Geomech. Eng.*, **13**(5), 793-807. http://doi.org/10.12989/gae.2017.13.5.793.

Tian, H., Wei, C., Wei, H. and Zhou, J. (2014), "Freezing and thawing characteristics of frozen soils: Bound water content and hysteresis phenomenon", *Cold Reg. Sci. Technol.*, **103**, 74-81. http://doi.org/10.1016/j.coldregions.2014.03.007.

Tomura, S., Maeda, M., Inukai, K., Ohashi, F., Suzuki, M. and Shibasaki, Y. (1998), "Characterization of adsorbed water on sepiolite.", *J. Clay Sci. Soc. Jpn.*, **38**(1), 1-9, http://doi.org/10.11362/jcssjnendokagaku1961.38.1.

Tone, K., Kamori, M. and Shibasaki, Y. (1993), "Adsorbed cations and water film thickness on the kaolinitic clay surface", *J. Ceram. Soc. Jpn.*, **101**(1180), 1395-1399, http://doi.org/10.2109/jcersj.101.1395.

Tuller, M. and Or, D. (2005), "Water films and scaling of soil characteristic curves at low water contents", *Water Resour. Res.*, **41**(9), http://doi.org/10.1029/2005WR004142.

Wang, H., Qian, H., Gao, Y. and Li, Y. (2020), "Classification and physical characteristics of bound water in loess and its main clay minerals", *Eng. Geol.*, **265**, http://doi.org/10.1016/j.enggeo.2019.105394.

Wang, W.Y., Li, A.M. and Zhang, X.M. (2011a), "DSC and SEM analysis on bound water characteristics in sewage sludge", *Adv. Mater. Res.*, **347**, 2085-2089. http://doi.org/10.4028/www.scientific.net/AMR.347-353.2085.

Wang, Y., Lu, S., Ren, T. and Li, B. (2011b), "Bound Water Content of Air-Dry Soils Measured by Thermal Analysis", *Soil Sci. Soc. Am. J.*, **75**(2), 481-487. http://doi.org/10.2136/sssaj2010.0065.

Watanabe, K. and Mizoguchi, M. (2002), "Amount of unfrozen water in frozen porous media saturated with solution", *Cold Reg. Sci. Technol.,* **34**(2), 103-110. http://doi.org/10.1016/S0165-232X(01)00063-5.

Xu, H., Shu, S., Wang, S., Zhou, A., Jiang, P., Zhu, W., Fan, X. and Chen, L. (2019), "Studies on the chemical compatibility of soil-bentonite cut-off walls for landfills", *J. Environ. Manage.*, **237**, 155-162. http://doi.org/10.1016/j.jenvman.2019.02.051.

Yeşilbaş, M. and Boily, J.F. (2016), "Particle size controls on water adsorption and condensation regimes at mineral surfaces", *Sci. Rep.*, **6**, 32136. http://doi.org/10.1038/srep32136.

Zeng, L., Xiao, L., Zhang, J. and Fu, H. (2020), "The Role of Nanotechnology in Subgrade and Pavement Engineering: A Review", *J. Nanosci. Nanotechnol.*, **20**(8), 4607-4618. http://doi.org/10.1166/jnn.2020.18491.

Zhang, J., Ding, L., Li, F. and Peng, J. (2020a), "Recycled aggregates from construction and demolition wastes as alternative filling materials for highway subgrades in China", *J. Cleaner Prod.*, **255**, 120223, http://doi.org/10.1016/j.jclepro.2020.120223.

Zhang, R., Wu, M., Kumar, P. and Gao, Q.F. (2020b), "Influence of loosely bound water on compressibility of compacted fine-grained soils", *Adv. Civ. Eng.*, **2020**, 1-14. http://doi.org/10.1155/2020/1496241.

Zymnis, D.M., Whitttle, A. and Germaine, J.T. (2019), "Measurement of Temperature-Dependent Bound Water in Clays", *Geotech. Test. J.*, **42**(1), 20170012, http://doi.org/10.1520/GTJ20170012.

CC