Measurement of unfrozen water in unsaturated soil with pulse NMR

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ABSTRACT

This study aims to reveal the effect of suction on the ice formation in unsaturated soil. Unfrozen water content was measured by a pulse NMR for the saturated and unsaturated silt prepared by vapor pressure technique. Based on the data, for the unsaturated frozen soil with the initial high water content, the unfrozen water content of the unsaturated soil has the same tendency as that of saturated soil. While, water rarely freezes in case of low water content. Water cannot freeze less than the water content corresponding to the beginning of residual water saturation for all conditions. Furthermore, the unfrozen water ratio of the unsaturated soil is higher than that of the saturated soil. Therefore, these results revealed that suction has strong influence on the ice formation in unsaturated soil.

Keywords: frozen soil, unsaturated soil, unfrozen water content

1 INTRODUCTION

Artificial ground freezing method has been used for construction purposes in Japan since frozen soils are stiff and low-permeable and stable. On the other hand, during freezing and thawing, frozen soils are unstable, and could cause geotechnical disasters in cold regions. One of the largest problems regarding freezing is frost heaving, which does severe damage to infrastructures. In addition, during snowmelt season, reduction of the bearing capacity and strength could cause the damage of infrastructures such as pavements and so on.

Unfrozen water in frozen soils has an impact on the strength, deformation, thermal conductivity and permeability (e.g. Takashi et al. 1981, Nagano and Ochifuji 1993, Tokoro et al. 2015). In addition, unfrozen water is necessary for frost heaving (Bescow, 1935). Therefore, it is essential to grasp the amount of unfrozen water content in frozen soils for understanding the behavior of frozen soils. Several methods to measure unfrozen water content have been developed, and the data has been shown in previous studies (e.g. Williams 1964, Ishizaki et al. 1996). However, most of the previous studies have focused on the unfrozen water content of saturated soils.

In this study, the unfrozen water content of the unsaturated soil and saturated soil was measured in order to clarify the characteristics of unfrozen water. Unsaturated soil specimens were prepared by using a vapor pressure technique and unfrozen water content was measured by using pulse NMR.

2 METHODOLOGY

2.1 Test material

The material used in this study is Fujinomori clay. Table 1 and Fig. 1 shows the physical properties and grain size distribution of Fujinomori clay respectively. Fujinomori clay is known as a frost susceptible soil, which means it contains unfrozen water content below 0°C.

| Sample name | $\rho_s$ (g/cm³) | $W_i$ (%) | $W_P$ (%) | $I_F$ |
|-------------|-----------------|-----------|-----------|------|
| Fujinomori  | 2.69            | 40.5      | 24.7      | 15.8 |

Fig. 1. Grain size distribution

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2.2 Pulse NMR

It is known that water partially exists as unfrozen water at subzero temperature. There are some techniques that determine the unfrozen water content in frozen soil. Among them, application of nuclear magnetic resonance is known to be an accurate and reliable method.

In this study, a NMR analyzer by Oxford Instruments (MQC) was used to measure unfrozen water content of saturated and unsaturated soils. The adopted operating frequency of the pulse is 23MHz.

Using this method, pulse application of a magnetic field induces voltage in its perpendicular direction, and the free-induction decay of proton is monitored. The decay is proportional to the amount of water in the specimen. As the decay in solid water (ice) is much faster than in liquid water, the decay curve can be readily decomposed into parts corresponding to each phase, and the amount of each phase can be quantified.

2.3 Procedure of measuring unfrozen water content

The samples were reconstituted from slurry with 80% water content under preconsolidation pressure of 500kPa. The specimens for pulse NMR test were trimmed from the preconsolidated soil. The specimen is cylinder specimen with height in 25 mm and a diameter in 17mm.

Unsaturated soil specimens were prepared by using vapor pressure technique. Vapor pressure technique uses the principle that the chemical potential of saturated aqueous solution balances with chemical potential of water in soil. When certain chemical potential of saturated aqueous solution is put in a closed vessel at constant temperature, the vapor in the vessel comes to be same value as that of the solution. Then, the enclosed space can keep relative humidity corresponding to the chemical potential. When specimen is put in the vessel, water in the soil finally balances the chemical potential of the vapor. Many saturated solutions have been used to obtain the SWCC, especially under high suction. Table 2 shows the solution used in this study. Fig.2 shows the soil water characteristic curve of Fujimori clay, obtained by combination of vapor pressure technique and pressure plate technique. Below the suction of 300kPa, it was obtained by pressure plate technique.

After equilibrium of the specimens was achieved, specimen was rapidly frozen in the freezer at temperature of -20 °C in order to prevent ice lenses from forming in the specimen. In this study, unfrozen water content was measured at the temperature of -0.2°C, -1°C, -5°C, and -10 °C. The specimen put into the freezer was translocated to the temperature controlled water bath of which temperature was -10 °C, and it was kept in the bath for more than 1 day to equilibrate. 24 hours later, the unfrozen water content was measured by pulse NMR.

Unfrozen water content was calculated by the method proposed by Akagawa et al (2012). FID curve contains both components of ice and unfrozen water. It was decomposed by the following equation.

\[ y = P_1 e^{\frac{-t}{T_1}} + P_2 e^{\frac{-t}{T_2}} \]

Where \( y \) is FID of ice and unfrozen water, \( P_1, P_2 \) are the constant values for unfrozen water and ice respectively, \( t \) is time, \( T_1 \) and \( T_2 \) are relaxation time of unfrozen water and ice.

Unfrozen water content \( w_u \) can be calculated by following equation, based on the assumption that FID intensity could increase with temperature decrease.

\[ w_u = 5wF_0/(5F_0(5F_3(5F_3+5F_1))(10-T_n)) \]

Where, \( w \) is water content at ordinary temperature, \( F_0 \) is the FID of water at each negative temperature, \( F_1 \) and \( F_3 \) are the FID at 10°C and 5°C, \( T_n \) is the temperature where unfrozen water content is measured.

After measuring the unfrozen water content, the specimen was put into the bath at which the temperature was changed to the next testing temperature.

The above procedure was repeated for different temperature of specimens to obtain the soil freezing curve (SWC).

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**Table 2: Saturated solution**

| Chemical Formula | Relative Humidity (%) | Suction (kPa) |
|------------------|-----------------------|--------------|
| K$_2$SO$_4$      | 98                    | 2830         |
| KNO$_3$          | 95                    | 6940         |
| KCl              | 85                    | 21900        |
| NaCl             | 75                    | 39000        |

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**Fig. 2. Soil water characteristic curve**
3 RESULTS

3.1 Soil Freezing Curve

Fig. 3 shows the unfrozen water contents of each initial water contents. Here, the initial water content $w_i$ means the water content before freezing, in other words, it is equal to the ice content by weight. The initial water content of 36.2% was fully saturated frozen soil. For the specimen with initial water content $w_i=36.2\%$, $w_i=16.8\%$, the unfrozen water contents on all temperatures have a common characteristic that unfrozen water contents have strong temperature dependency. Namely, in subzero temperature, water remains liquid, and unfrozen water decreases with temperature decrease. While, for the initial water content of 8.9%, 8.1% and 6.8%, unfrozen water content rarely changes with the decrease in temperature.

Unfrozen water content ratio, calculated by dividing unfrozen water content by initial water content, is shown in Fig. 4. For the high initial water content such as $w_i=36.2\%$ and $w_i=16.8\%$, the ratio decreases with temperature decrease. For the high initial water content such as $w_i=8.9\%$, $w_i=8.2\%$ and $w_i=6.8\%$, the ratio is over 0.9, which means more than 90% of water exists as unfrozen water at any temperature. Compared unfrozen water content ratio among the temperatures, they are ranked in ascending order of the amount of initial water content at all temperatures. It is inferred from this data that the initial water content or suction applied to the specimen before freezing have influence on the unfrozen water content of unsaturated frozen soil.

Fig. 5 shows the relation between the initial water content and the unfrozen water content. The soil water characteristics curve obtained by the vapor pressure technique is also shown in the figure to evaluate the effect of suction on the unfrozen water content in the unsaturated frozen soil. The unfrozen water content of unsaturated frozen soils has the same tendency as saturated frozen soils; it declines with the reduction of temperature. Furthermore, when the unfrozen water content below 8%, corresponds to beginning of the water content of residual water saturation, the water exists exclusively as unfrozen water. This indicates high suction before freezing which prevents the phase change from liquid to ice. According to the findings, suction could have a strong influence on the ice formation in frozen soils.

3.2 Comparison with SFC and SWCC

Miller (1965) proposed the similarity between soil water characteristic curve and soil freezing curve. Black and Tice (1988) revealed the similarity by comparing the soil freezing curve obtained by pulse NMR with SWCC.

The pressure difference between air and water, namely matric suction of ice-free soil, is expressed as Equation (3).

$$
\phi = u_s - u_w
$$

Where $u_s$ and $u_w$ are air pressure and water pressure respectively. In a similar way, for frozen soil, the pressure difference between ice and water is expressed as follows.

$$
\phi = u_i - u_w
$$

Where $u_i$ and $u_w$ are ice pressure and water pressure respectively.

In case of the same silt or clay with same dry density, if the unfrozen water content in frozen soil is equal to the water content in unsaturated soil, water distribution is the same in soils.
unfrozen water contents at the temperature of -5 °C and -10 °C get to be close to the water contents on the SWCC under high suction as with the temperature of -0.2°C and -1°C. This indicates that water in residual water saturation cannot freeze since the water exists like thick film and the magnitude of water pressure is high.

There are few researches about unfrozen water in unsaturated soil, so that few prediction model that can express SFC of unsaturated soil. Considering that water rarely freezes below the water content corresponding to residual water saturation, there is a possibility that the equation proposed by Black and Tice (1988) can be extended to unsaturated frozen soil.

4 CONCLUSIONS

- The amount of unfrozen water of unsaturated soil strongly depends on the initial water content. For the soil with high water content, the tendency is similar to the saturated soil. On the other hand, for the soil with low water content, the amount of water content does not change even though the temperature decreases.
- The unfrozen water content ratios of each initial water contents are ranked in ascending order of the amount of initial water content at all temperature.
- When the unfrozen water content below 8%, corresponds to the water content of residual water saturation, the water exists exclusively as unfrozen water.
- According to the comparison of SFC with SWCC proposed by Black and Tice (1988), Fujinomori clay is classified into the soil where capillary space is dominant.
- Considering that water rarely freezes below the water content corresponding to residual water saturation, there is a possibility that the equation proposed by Black and Tice (1988) can be extended to unsaturated frozen soil.

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