Experimental study on the variation of unfrozen water content and pore water in unsaturated soil during thawing process

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Abstract: Based on nuclear magnetic resonance (NMR) technique, the variation characteristics of unfrozen water content and the occurrence law of unfrozen water in pore structure of silty clay, red clay and remolded mudstone were analyzed by thawing experiments under different temperature gradients. The results show that the thawing process can be divided into negative temperature without phase change temperature rise stage, violent phase change melting stage and positive temperature without phase change temperature rise stage, and there is no superheating phenomenon corresponding to the supercooling phenomenon. The ice in the small pores continued to thaw during the whole test process. The ice stored in the macropores began to thaw in 30mins, and basically thawed in 40mins. The temporal order of pore ice melting is caused by the difference of hydrothermal kinetic potential energy. And the temperature gradient has no significant effect on the sequence of pore ice melting.

Keywords: unsaturated soil, frozen soil, nuclear magnetic resonance, unfrozen water content, micropore development, pore size distribution

1. Introduction
Climate warming caused by the greenhouse effect has become a global environmental problem of worldwide concern. As the temperature rises, the permafrost and seasonal
permafrost thaw slowly, and the temperature and unfrozen water content change correspondingly, which greatly affects the mechanical properties and hydrological characteristics of the soil. Permafrost is a complex porous medium composed of solid particles, liquid water, gas and ice, and the content of various components is an essential index for thermal calculation of permafrost, so it is particularly necessary to study the distribution curve of pore radius from the microscopic pore structure level[1-4].

In recent years, many scholars at home and abroad have made in-depth studies on the changes of unfrozen water content and microstructure of unsaturated soil during freeze-thaw process, and a series of important achievements have been made. Xiao Liang et al. converted NMR $T_2$ distribution into pore-throat radius distribution by piecewise power function, and obtained the relationship between pore size and pore throat size[5]; Wang Kewen et al. studied the NMR properties of saturated rocks and the influence of pore structure on the $T_2$ distribution curve. With the increase of pore-throat radius, the $T_2$ distribution moved to the direction of prolonged relaxation time and the peak intensity increased[6]. Westphal et al. divided the pores in saturated silicate rocks into two main types by $T_2$ cut-off values and proposed appropriate $T_2$ cut-off values for different types of carbonate rocks[7]. Yan Weichao et al. proposed a rock porosity testing method based on dry rock detection, which broke through the limit of echo spacing and detected micropore signals. Yao Yanbin et al. proposed a permeability model and pore structure model that can effectively estimate the pore size distribution of coal[9]; Liu Yonggian et al. conducted NMR tests on unmodified silt soil and soil samples after triaxial tests, and analyzed the variation rules of pore distribution, size and structural parameters of both[10]. Wang Ping et al. analyzed the development of rock porosity of argillaceous shale under different immersion times through NMR experiments[11]. In addition, the change of unfrozen water content in frozen soil with temperature and the transformation of solid and liquid water lead to the corresponding change of soil properties. The main testing methods of unfrozen water include calorimetry, pulsed nuclear magnetic resonance (NMR), differential scanning calorimetry (DSC), frequency-domain reflection (FDR), frequency-domain propagation (FDT), time-domain reflection (TDR) and time-domain propagation (TDT), etc[12-15]. The content of unfrozen water mainly depends on three factors: soil quality, external conditions and the history of freezing and thawing[16]. Leng Yifei analyzed the influence of negative temperature, soil type and initial water content on unfrozen water content and its change rule[12]. Although many studies have shown the effects of freeze-thaw process on various properties of soil, they mostly focus on mechanics [17-18]. Therefore, it is of great significance to analyze the characteristic curve of unfrozen water content from the Angle of pore diameter and the influencing factors of unfrozen water content from the Angle of micro view. However, there is a lack of research on this at home and abroad at present.

During freeze-thaw process, unsaturated soil often causes changes in its internal pore distribution and structure, which is difficult to be studied by conventional experimental means. In this paper, the properties of unfrozen water in unsaturated silty clay were studied by low-field NMR method. Combined with the measurement results of unfrozen water content and pore size distribution results, the variation characteristics of unfrozen water content in soils under different temperature gradients were analyzed. At the same time, the evolution law of unfrozen water content curve in frozen soil was analyzed from the Angle of pore size
distribution curve.

2. Engineering background

2.1. Experimental materials and procedures

The three soil samples used in this experiment are the silty clay in Beijing, the red clay in Shaanxi and the mudstone (mudstone-hard soil) in Dabancheng, Xinjiang. The basic physical parameters and particle size distribution of the three types of soils are shown in Table 1. The fine grain content of silty clay, red clay and remolded mudstone are 81.81%, 89.29% and 93.82% respectively. First, all kinds of soil will be obtained through 0.5mm sieve, and at 105°C temperature drying 12h until the weight does not change. Then spread it on the mixing tray, spray a small amount of distilled water with a watering can several times, then sprinkle another layer of drying soil, and repeat the process. Sealed with plastic wrap and left standing for 24h to ensure even water migration in the soil sample. The configured soil samples will be pressed into Ф20mm×30mm, 20mm×20mm, and 20mm×10mm ring tubes for 3 times to make standard samples, and the individual quality will be roughly equal to the control. The initial water content of silty clay, red clay and remolded mudstone samples were 22%, 28% and 38%, respectively. Eight samples were prepared for each specification of different soil quality, and a total of 72 samples were prepared. In order to eliminate the influence of ferromagnetic materials on the uniformity of the main magnetic field, Teflon band was wrapped and then placed in the cryogenic cold bath with cryogenic solution ethylene glycol-frozen to -40°C. When the internal and external temperature of the soil reached the same level, it was quickly taken out of the cryogenic cold bath and placed in the NMR sample tube, and the parameters of NMR signal acquisition were set. The free induction attenuation (FID) curve was acquired, and then the \( T_2 \) distribution was obtained by Fourier transform inversion software, and the NMR signal data were saved. Repeat the above steps and measure all temperature points in the heating process until the test is completed. The constant temperature system of the magnet and the power supply of the RF unit were turned on 1 d in advance to ensure the stability and uniformity of the magnetic field of NMR, and no secondary disturbance was carried out on the sample during the experiment to ensure the accuracy of the test. By analyzing the NMR detection signals of samples, the pore measurement results and unfrozen water content at each temperature point can be obtained. Then, the influence law of thawing process on the pore structure of soil and the change trend of unfrozen water content -- temperature curve under the influence of different gradients are analyzed.

Table 1. Basic physical parameters and particle composition of the soil specimens

| Soil       | Plastic limit (%) | Liquid limit (%) | \( I_p \) | Specific surface area \( (m^2\cdot g^{-1}) \) | Particle composition (%) |
|------------|-------------------|------------------|-----------|--------------------------------------------|-------------------------|
|            |                   |                  |           |                                            |                         |
| Silty clay | 9.85              | 23.98            | 14.13     | 142.1                                      | ≤0.002: 3.44, 0.002–0.005: 9.05, 0.005–0.075: 69.32, >0.075: 18.19 |
| Red clay   | 9.24              | 28.75            | 19.51     | 173.6                                      | ≤0.002: 4.57, 0.002–0.005: 11.43, 0.005–0.075: 73.29, >0.075: 10.71 |
2.2. Experimental apparatus and principle

The main equipment of the test is nuclear magnetic resonance testing system, low temperature constant temperature tank, electronic balance, TDS-303 data acquisition instrument, drying box, etc. The nuclear magnetic resonance test system is the MicroMR12-025V nuclear magnetic resonance core analyzer produced by Suzhou NIUMAG Electronic Technology Co., Ltd. The magnetic field intensity of the equipment is 0.28±0.05T, the main frequency of the instrument is 12MHz, the RF pulse frequency is 1.0 ~ 49.9MHz, the diameter of the probe coil is 25.4mm, and the temperature of the magnet cabinet is 32°C. The experimental equipment is shown in figure 1.

![Figure 1. Experiment equipments](image)

(a) samples and sensors (b) low temperature tank (c) data acquisition instrument (d) nuclear magnetic resonance testing system

Nuclear magnetic resonance (NMR) is a testing technique to obtain the information of hydrogen nuclei by the interaction between hydrogen nuclei and external magnetic fields in the sample. During the test, a certain frequency RF pulse is applied after the sample is placed in the main magnetic field. The spin hydrogen nuclei in the sample are magnetized and absorb certain energy, which deviates from the equilibrium state. When the radiofrequency is terminated, the magnetized hydrogen nucleus gradually reaches equilibrium state. The time that the hydrogen nucleus takes from the disequilibrium state to the equilibrium state is the relaxation time [19-21].

NMR is a powerful tool to study the structure of porous media, which can provide information related to the pore structure of porous media, such as porosity, pore distribution, permeability, water saturation and so on. The main source of low field NMR signal is hydrogen in water. When the pores in the substance are filled with water and the substance itself does not provide NMR signal in low field NMR, the porosity of the substance can be
converted by the intensity of NMR signal obtained. Due to the higher S/V value of the smaller pores, the smaller the pores are, the more frequent the collision between hydrogen nuclei and the pore wall is, the faster the energy loss is, and the shorter the corresponding transverse relaxation process is. Therefore, $T_2$ is directly proportional to the pore radius[22]. This relationship can be used to study the pore size and its connectivity in soil samples by NMR experiment.

The relaxation time $T_2$ of general materials is expressed as:

$$\frac{1}{T_2} = \frac{1}{T_{2,\text{free}}} + \frac{1}{T_{2,\text{surface}}} + \frac{1}{T_{2,\text{diffusion}}}$$

$$= \frac{1}{T_{2,\text{free}}} + \rho_2 \left( \frac{S}{V} \right)_{\text{pore}} + \frac{D(\gamma GT_z)^2}{12}$$

(1)

Where, $T_{2,\text{free}}$ is the free relaxation time of the fluid, which is determined by the physical properties of the fluid[23]; $T_{2,\text{free}}$ is the surface relaxation time of the fluid, which is calculated by the ratio of surface relaxation intensity ($\rho_2$) and pore surface area ($S$) to volume ($V$); $T_{2,\text{diffusion}}$ is the diffusion relaxation time of the fluid, which is calculated from the diffusion coefficient($D$), magnetic field intensity($G$), magnetic spin ratio($\gamma$) and the sequence parameter($T_E$) at the time of measurement.

In calculation, $T_{2,\text{free}}$ and $T_{2,\text{diffusion}}$ can be ignored, and relaxation time is mainly determined by $T_{2,\text{surface}}$. Therefore, Equation (1) can be simplified and the relationship between pore distribution of excavated samples and relaxation time $T_2$ can be obtained as follows:

$$\frac{1}{T_2} = \rho_2 \left( \frac{S}{V} \right)$$

(2)

Where, $\rho_2$ is the surface relaxation strength, which is a constant that changes with the change of material properties; ($S/V$) is the ratio of pore surface area volume, which is $(3/R)$ when using the sphere model, and $(2/R)$ when using the cylinder model.

Therefore, with the help of surface relaxation rate, the corresponding relationship between relaxation time T2 and pore radius can be established. Moreover, according to the NMR relaxation mechanism, the position of the relaxation time on the $T_2$ distribution curve varies with the different pore types of the fluid in the medium. According to the distribution of peak signal of $T_2$ spectrum, the development characteristics of pores in soil samples can be judged. The area of the peak reflects the number of pore volumes corresponding to a certain aperture range, the width of the peak reflects the sorting of the corresponding pores, and the number of the peak reflects the continuity of all levels of pores.

3 Experiment results and analysis

3.1 Uniformity of temperature field of small volume sample

When the temperature difference between the internal temperature and the surface temperature of the object is not large, this kind of unsteady heat conduction process can be treated as: the temperature distribution in the object has no relationship with the spatial position coordinates, but only changes with time. In addition, the internal temperature distribution of the object is uniform and equal, and the temperature of any point can represent the overall temperature, and the physical parameters such as the heat capacity and mass of the
object are concentrated at this point, which is called lumped parameter method [24]. In subsequent experimental studies, the Biot Number $Bi$ (the dimensionless number representing a physical phenomenon or physical process, also known as the criterion number) needs to be calculated first:

$$Bi = \frac{hl_e}{\lambda_s} = \frac{\frac{h e}{\lambda s}}{\frac{h}{e}} = \frac{\text{Heat conduction resistance inside the solid}}{\text{External heat transfer resistance of solid}} \quad (3)$$

Where, $h$ is the heat transfer coefficient of the surface of the object (W·m⁻²·K⁻¹); $l_e$ is the reference size, for infinite flat wall (thickness is $2\delta$), $l_e=\delta$; $l_e=d/2=R$ (radius) of an infinitely long cylinder or sphere; $\lambda_s$ is the thermal conductivity of the object (W·m⁻¹·K⁻¹). When $Bi \to 0$, the thermal conductivity resistance inside the solid can be approximately ignored. At any moment, the internal temperature of the object is uniformly distributed, and with the change of time, the overall temperature approaches to the temperature of the fluid. As $Bi \to \infty$ the heat transfer resistance outside the solid can be approximately ignored. At the initial moment, the surface temperature of the object is the temperature of the fluid. As time goes on, the temperature at various positions inside the object approaches to the temperature of the fluid. When $Bi$ is a finite value, it indicates that external heat transfer resistance and internal heat conduction resistance cannot be ignored, and the temperature field in the object is between the above two conditions. If the lumped parameter method satisfies the applicable conditions, the internal thermal resistance of the solid is required to be ignored. In engineering practice, it is generally believed that the excess temperature error at each point of the object is no more than 5%, that is, the characteristic number $Bi \leq 0.1$. Here, a silt cylindrical sample with a height of $H=30\text{mm}$ and a radius of $R=10\text{mm}$ is taken as an example to calculate the Biot Number $Bi$.

Sample volume:

$$V=\pi R^2 H=3.14\times0.01^2\times0.03=9.42\times10^{-6}\text{m}^3 \quad (4)$$

Sample surface area:

$$A=2\pi R^2+2\pi RH=2\times3.14\times0.01^2+2\times3.14\times0.01\times0.03=2.512\times10^{-3}\text{m}^2 \quad (5)$$

reference size:

$$l_e=\frac{V}{A}=\frac{9.42\times10^{-6}}{2.512\times10^{-3}}=0.00375\text{m} \quad (6)$$

According to the measured experience, the heat transfer coefficient $h$ of the surface of the soil and the air is between 12-15 (W·m⁻²·K⁻¹), and the thermal conductivity coefficient of the soil sample at normal temperature is 1.62 (W·m⁻¹·K⁻¹). The characteristic number of samples can be obtained as follows:

$$Bi=\frac{hl_e}{\lambda_s} = \frac{0.00375	imes15}{1.62} = 0.0354 \leq 0.1 \quad (7)$$

It can be seen that the external heat transfer resistance of soil is much greater than the internal thermal conductivity resistance of soil. The design of the volume parameters meets the theoretical requirements, and the samples as a whole meet the basic assumption of the uniformity of the internal temperature field. Therefore, the samples can be used as the concentration point for research[25].
3.2 Calculation of unfrozen water content under different temperature gradients

Due to the linear relationship between the pore water NMR signal and the temperature in the positive temperature range, the measured data in the positive temperature range can be used to fit the paramagnetic linear regression line. When the soil temperature is positive, the NMR semaphore decreases linearly with the increase of temperature. However, when the temperature drops to negative temperature, as the temperature continues to drop, part of the free water begins to freeze. The signal from solid ice does not show up in the NMR, so the signal begins to decline, but the rate of decline gradually decreases until it stabilizes.

According to the decrease of the NMR signal conversion cannot frozen water content of soil samples, by is reverse extend the temperature range of paramagnetic linear regression line (see Figure 2.), negative temperature regions in a certain temperature of frozen water content is equal to the temperature measured signal strength value multiplied by the initial moisture content of a divided by the temperature signal strength b value shown in the tropic of cancer. After freezing, due to the adsorption of the capillary and particle surface, with the decrease of temperature, there is always a part of liquid water that does not freeze into ice, and the dynamic balance between this part of water and ice is always maintained. And can be expressed in the following formula[8]:

\[ \omega_u = a \theta^b \]  

(8)

where: \( \omega_u \) is unfrozen water content; \( \theta \) is the negative absolute value of temperature; a and b are empirical values related to soil quality. In this experiment, the data of three different soil samples were fitted, and the exponential relationship between unfrozen water content and temperature after the initial freezing temperature was well verified by drawing the curve as shown in figure 3, which indicated that the measurement of unfrozen water content by NMR technology was reliable.

Figure 2. Calculation of unfrozen water content
Figure 3. Unfrozen water content-temperature curve

Figure 4. shows the unfrozen water content-temperature characteristic curve of silt samples with dry density $\rho_d=1.44\ \text{g/cm}^3$. As can be seen from the figure, the soil goes through three stages in the thawing process. In the stage $I$ (-40°C~0.3°C), the content of pore water is basically stable, and the solid ice basically does not change phase. In the lower temperature range, the content of unfrozen water increases with the rise of temperature, which belongs to the stable stage. At the stage $II$ (-0.3~0°C), with the increase of temperature, the ice thaws into water, and the pore water content in the soil increases rapidly and the change is more dramatic, which belongs to the rapid rise stage. At the stage $III$ (after 0°C), with the continuous rise of temperature, the unfrozen water content in the soil tends to be stable, and the ice in the void basically thaws into water.

Due to space limitation, only silty clay samples are taken as an example for analysis. Under different temperature gradients, the unfrozen water content increases with the increase of the specific surface area of soil particles, and the corresponding soil particle surface adsorption is stronger, and the thawing point of solid ice is lower. In addition, it can also be seen from the figure 4 that the unfrozen water content of the soil sample is basically stable in the temperature range below -20°C. As the volume of the sample decreases, the unfrozen water content of the soil sample increases somewhat in the stage $I$ of thawing. Under the same negative temperature environment, unfrozen water content of red clay and mudstone samples fluctuated in the range of 7.5%~8.3% and 4.7%~5.5%, among which the remolded...
mudstone contains more fine particles, which is more stable at the stage of thawing compared with the other two kinds of soil. The variation of unfrozen water content is similar to that of silt in the tests of various samples.

3.3 Relationship between T2 relaxation curve and pore structure

Based on the relationship between relaxation time $T_2$ and pore radius, the $T_2$ distribution curve is transformed into pore component curve. The horizontal axis of the pore component curve represents the pore radius; the vertical axis represents the percentage of the pores corresponding to this size in the total internal pores of the sample, namely the porosity component; the area enclosed by the pore component curve and the horizontal axis is the total porosity of the sample.

![Figure 5. T2 distribution of red clay](image)

![Figure 6. Pore size distribution curve during thawing](image)

Figure 5 shows the $T_2$ distribution curve of red clay during freezing. Free water (including capillary water and gravity water) occurs in large pore size, and bound water occurs in particles in small pore size. Under the influence of binding action, bound water has lower surface free energy than free water and is more difficult to freeze during cooling.

Figure 6 shows the pore distribution curve of the thawing process. In order to avoid repeated analysis, only the red clay sample is taken as an example. The variation and distribution of the curve peak area during the thawing process reflects the variation of the amount of unfrozen pore water in the soil. As can be seen from figure 5, large pores and small pores mainly exist between the peak area of 0.001-1μm, and the peak area of pore size of 0.001-1μm is larger than that of 0.11-10μm. The first peak of the spectrum plane is the
corresponding small-size pores that account for the largest proportion of the total area, indicating that micro-pores account for the vast majority.

In the early stage of thawing, the increase of large pores is not large, but the pore water of small pores increases rapidly. When the temperature rises to thawing temperature, the unfrozen pore water in the soil increases greatly, and the increase rate of the large pore is higher than that of the small pore, and the unfrozen pore water with the pore size of 0.01µm increases significantly. As the temperature continues to increase, the increase of macropores is small. The peak area of the curve increases with the thawing process, showing a "overall right-shift" pattern, and the increase of pore water starts from small pores. During the thawing process, the ice in the small pores continues to melt. At 30 minutes, the ice in the macropores began to thaw, and at 40 minutes, the ice in the macropores is basically thawed.

Conclusion

1) The melting process of silty clay, red clay and remolded mudstone samples with different sizes all went through three stages. The stage I is a temperature rise stage with negative temperature and no phase change. Most of the water in the soil sample existed in the form of ice phase, but there was still a small part of the bound water that was not frozen. The stage II is a stable melting stage, in which most of the ice melts into liquid water when the temperature changes by 1℃. The stage III is the positive temperature and no phase change temperature rise stage, the temperature rises rapidly in this stage, all the ice in the pores melt into water, and the unfrozen water content of the soil is equal to the initial water content.

2) The unfrozen water content of the three kinds of soil samples under different temperature gradients is basically stable in the temperature range below -20℃. With the decrease of the sample volume, the unfrozen water content of the three kinds of soil samples at the stage I of thawing increases, but the numerical difference is not significant. The remolded mudstone contains more fine particles, which is more stable than the other two types of soil at the stage I of thawing.

3) Combined with the T2 distribution curve and pore size distribution curve, the distribution of unfrozen water in the pores under different temperature gradients during the thawing process is analyzed from the microscopic point of view. At the initial stage of the experiment, about 95% of the unfrozen water in the soil existed in small pores. During the thawing process, the ice in the small pores continues to melt. At 30 minutes, the ice in the macropores began to thaw, and at 40 minutes, the ice in the macropores is basically thawed. Although there are differences in the water potential energy of different pore sizes, the difference of temperature gradient does not significantly affect the order of ice thawing in pore sizes.

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