



Unfrozen Water Content and Developmental Mechanisms at the Structure–Soil–Stone Mixture Interface in Seasonally Frozen Regions

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Abstract. The rise in temperature induces alterations in the unfrozen water content(ω_u) at the soil-rock mixture and concrete interface, leading to a subsequent deterioration in interfacial strength. In this study, rough concrete surfaces were fabricated via 3D printing technology, and composite specimens comprising soil-rock mixtures and concrete with varying stone contents were prepared. The nuclear magnetic resonance (NMR) experiments were conducted to investigate the variation in unfrozen water content at the interface during the positive melting phase. The T_2 cut-off value was subsequently determined, allowing for the classification of pore water types, followed by an analysis of the developmental patterns of each type. The results reveal that the interfacial ω_u undergoes three distinct phases of change in response to varying rock content during the melting process. Analysis of the T_2 distribution identified 4.5 ms as the cut-off value separating bound water from capillary water in this specimen. The development of both bound and capillary water predominantly occurs below 0°C, with the growth rate of capillary water exceeding that of bound water. However, the content of bound water consistently remains higher than that of capillary water throughout the process.

Keywords: Soil-rock mixture; Structure interface; NMR; Unfrozen water; Melt; 3D printing technology

1 Introduction

In the seasonal freezing zone, as the temperature increases, internal ice crystals within the soil-stone mixture melt, water migrates, and the bonding arrangement of soil and stone particles, along with their contact with structural surfaces, undergo changes. These alterations lead to the deterioration of the strength at the soil-stone mixture-structural interface, resulting in thaw settlement, landslides, and other geohazards. The unfrozen water (ω_u) in permafrost plays a critical role in processes such as water-heat transfer, frost heave, thawing, and mechanical degradation of permafrost ^[1]. Therefore, it is essential to quantify unfrozen water content at various temperatures and analyse these changes to better understand the development patterns of different

categories of pore water. Feng et al.^[2] examined the available techniques for measuring ω_u in permafrost, categorizing them into five major techniques: pressure, radiometric, electromagnetic, thermal, and acoustic methods. The Nuclear Magnetic Resonance (NMR) method employed in this experiment utilizes the distinct relaxation times of hydrogen nuclei in a magnetic field to generate signals of varying intensities, which correspond to the moisture content in the specimen. This method was first introduced by Tice et al.^[3] for assessing ω_u in permafrost, significantly improving test accuracy. Despite the high cost and lengthy duration of NMR tests, its strong anti-interference capacity, non-destructive nature, and high precision make it one of the most advantageous techniques for assessing unfrozen water in permafrost^[4]. The soil-rock mixture is a highly heterogeneous and loosely structured geological body, composed of a complex medium with a highly varied particle composition^[5]. Its internal structure and mechanical properties become even more complex during freeze-thaw cycles^[6]. Additionally, the strength of the soil-rock mixture-structure interface is strongly influenced by its ω_u . This study utilized 3D printing to create rough concrete surfaces that closely simulate real-world engineering conditions, and to fabricate soil-rock mixture-concrete specimens with varying stone contents. NMR tests were conducted to obtain T_2 curves at different temperatures across all interfaces, allowing for the investigation of unfrozen water content changes during the thawing stage. The T_2 cut-off value was determined to classify the different types of pore water, which served as the basis for analyzing the developmental patterns of each pore water type. The findings from this study offer new perspectives for the mitigation of thawing-related subsidence and other hazards in cold-region engineering projects, both for future developments and existing infrastructure.

2 Materials and Test Methods

2.1 Specimen Preparation for Structural-soil-rock Mixture

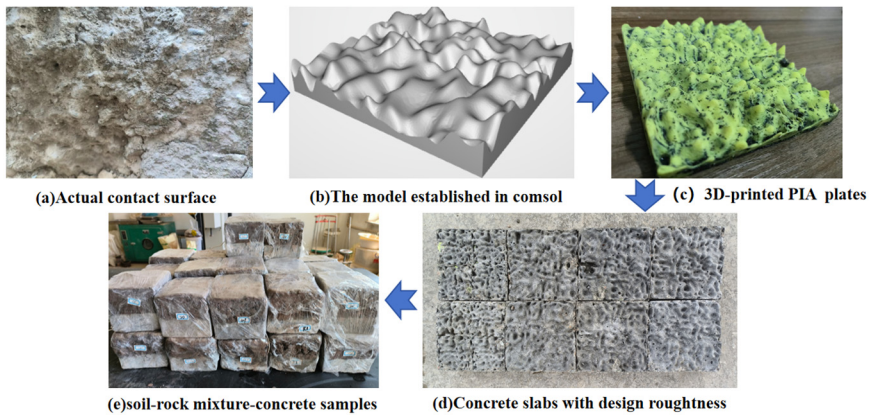


Fig. 1. Preparation of structural-soil-rock mixture specimens using 3D printing technology

As shown in Fig. 1, the structure-soil contact surface from the actual project was first collected, and a model was generated using COMSOL and subsequently printed with 3D printing technology. A concrete specimen with a designed rough surface, measuring 100 mm × 100 mm × 50 mm, was produced by casting with the model. After the curing process was completed, a pre-configured soil-rock mixture was added and compacted in three stages to form the structure-soil-rock mixture specimen.

2.2 NMR Experimental Equipment and Principle

NMR test was performed using the MacroMR12-150H-I NMR test equipment, and the transverse relaxation time (T_2) for porous media, such as soil-stone mixtures, can be expressed as follows:

$$\frac{1}{T_2} = \rho \left(\frac{S}{V} \right)_{\text{pore}} = \rho \left(\frac{F}{R} \right)_{\text{pore}} \quad (1)$$

where ρ is the surface relaxation rate, $(S/V)_{\text{pore}}$ is the ratio of pore surface area to pore volume, F is the geometry factor, and R is the radius of the pore. It can be concluded that the larger the T_2 value, the larger the corresponding pore size and the more unfrozen water content in the pore. The intensity of the NMR signal for the T_2 value is proportional to the number of H atoms, which represents the liquid water content at that corresponding pore size. A paramagnetic linear regression line^[7] was plotted using measured data in the positive temperature zone to calculate the unfrozen water content ω_u . The calculation method is as follows:

$$\omega_u = \frac{b}{a} \omega \quad (2)$$

where ω is the initial moisture content of the soil sample, b is the NMR signal intensity measured at the negative temperature, and a is the NMR signal intensity corresponding to the paramagnetic linear regression line at that temperature.

3 Result and Discussion

3.1 Temperature Dependence of Interfacial ω_u

As can be seen from Fig. 2, ω_u of the interfaces with different rock contents during the thawing phase varies in approximately the same three stages. The first phase is the stable melting stage (below -6°C), where ω_u gradually increases with a slow rise in temperature, primarily due to the melting of bound water at the interface. The second phase is the rapid melting stage (-6°C to 0°C), during which ω_u rises sharply in response to temperature increases, showing a significant 7% to 9% rise from -6°C to 0°C . Interestingly, the interface with 60% rock content displays a lower ω_u curve in comparison to interfaces with lesser rock content throughout the first two stages. This phenomenon can be ascribed to the abrupt rise in pore numbers at the 60% rock content interface, which consequently results in a marked increase in capillary water con-

tent and a sharp reduction in bound water content. The final phase, the post-thaw stabilization stage (above 0°C), ensues once all ice crystals at the interface have transitioned into liquid.

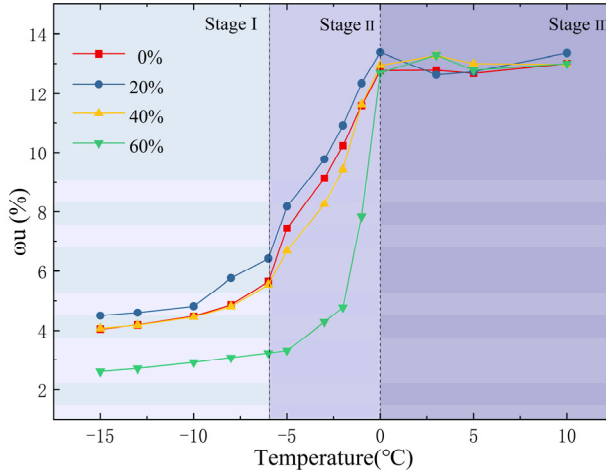


Fig. 2. Variation curves of ω_u at interfaces with different rock contents

3.2 Classification of Unfrozen Water and Their Developmental Regularities at the Interface

The T_2 spectrum reveals a clear T_2 cut-off value (Division of relaxation times between different pore waters on the T_2 spectrum) between bound water and capillary water. This cut-off value remains consistent across soil-stone mixtures with varying stone contents but identical compositions, as analyzed based on the methodology outlined in a related study^[8]. When all the unfrozen water in permafrost is adsorbed, its content exhibits minimal variation with temperature^[9]. Examining the T_2 curve as Fig.3 (a), it is evident that when the temperature exceeds -5°C, both the growth in peak area and the rightward shift of the curve become more pronounced as the temperature increases. Conversely, when the temperature falls below -5°C, these changes are significantly less pronounced. Thus, the maximum T_2 value at -5°C, measured at 4.5 ms, was identified as the cut-off value distinguishing bound water from capillary water within the specimen. The T_2 value reflects the pore size within the soil, and as the temperature rises, the T_2 curve shifts rightward, indicating that unfrozen water development progresses from smaller to larger pore spaces, as the ice-water phase transition in smaller pores occurs at lower temperatures^[10].

By calculating the peak areas corresponding to bound water and capillary water based on the T_2 cut-off value determined in the previous text, the content of bound water and capillary water can be obtained. The changes and percentages of bound and capillary water content at the interface during the melting process were plotted, as shown in Fig.3 (b). The figure illustrates that the development of both bound and capillary water predominantly occurs below 0°C, with minimal change observed at

higher temperatures. Below 0°C, as the temperature increases, the bound water increases gradually, with the capillary water showing a relatively faster growth rate, which further accelerates as the stone content increases. According to the Gibbs-Thomson effect^[11], larger interfacial pore apertures lead to higher ice-water phase transition temperatures, meaning that the capillary water transitions at a higher temperature than bound water. At lower temperatures, bound water begins to form earlier than capillary water, resulting in a significantly higher bound water content below 0°C. As the temperature rises above freezing, the interfacial pores expand, leading to an increase in capillary water content, although bound water still remains the dominant fraction.

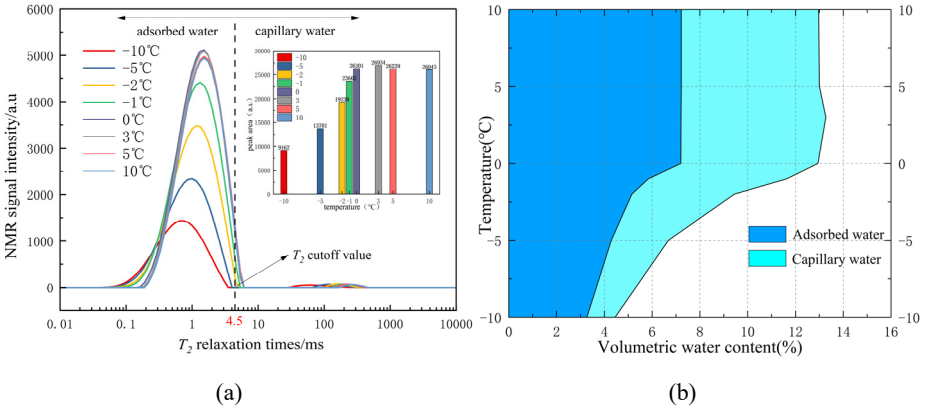


Fig. 3. (a) T_2 curve and T_2 cut-off (b) Temperature dependence of different pore water contents at the interface (Example of 40% stone content)

4 Conclusion

In this study, we constructed a soil-stone mixture-structure interface that more accurately reflects real-world conditions using 3D printing technology. Based on the NMR inversion of unfrozen water content and its distribution at the interface. This study is important for the prevention and control of thawing and subsidence and other diseases in cold zone engineering, the following conclusions were drawn:

- The changes in ω_u at the interface during the thawing process can be categorized into three distinct phases: a stable thawing phase, a rapid thawing phase, and a stable post-thawing phase.
- The unfrozen water present at the interface primarily exists as bound and capillary water, with the T_2 cut-off value for distinguishing between these types set at 4.5 ms. The T_2 curve shifts to the right, indicating that the development of unfrozen water progresses from smaller to larger pore spaces.
- Both bound and capillary water begin to develop before reaching normal temperatures, with capillary water exhibiting a faster growth rate compared to bound wa-

ter, and the bound water content consistently exceeds the capillary water content throughout the melting stage.

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