Using an NMR Device to Determine Unfrozen Water Content in Frozen Soil

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Abstract

The overall goal of our research project is to study the unfrozen water mass and mobility in frozen soils. During this project, frozen samples of standard clays with different adsorbed cations will be analyzed to determine their surface potential, micro-fabric, and how they interact with unfrozen water. The amount of unfrozen water content at sub-freezing temperatures is measured using a nuclear magnetic resonance (NMR) device. Before using the NMR to test the unfrozen water content, we developed a system to control and stabilize the temperature of the soil sample during the test. We determined that the optimal sample length detected by the NMR is 3.5 cm. Nine duplicate silt samples each with a different moisture content were prepared and tested using the NMR. The result demonstrated a linear relationship between the moisture content of the silt samples and the corresponding NMR signal intensities, thus validating the NMR approach. Future test will be conducted on frozen cation-exchanged clay samples to determine their unfrozen water contents as a function of temperature.

Introduction

A key element in understanding soil freezing and thawing processes is the ability to predict the mass fraction and mobility of unfrozen water (that is, how much water remains liquid at below-freezing temperatures in a soil-water mixture, and how does it move) within frozen soils. This research will use a combination of measurement techniques to quantify relationships among hypothesized key variables in the soil freezing and thawing processes. One of the steps in the research (and the subject of this poster) is developing a repeatable method to use an NMR device to detect the amount of unfrozen water in saturated soil at below-freezing temperatures.

Preparation and Calibration of NMR

In order to use the NMR to test the unfrozen water content of soil samples, the temperature of the soil sample should be known and stable during testing. A system shown in Figure 1 was developed to control the temperature of the soil samples. Lab tests were conducted to study the relationship between the temperature in the cooler and the temperature in the cooling bath. We determined the relationship between the temperatures in the cooler and the cooling bath by recording both temperatures, and plotting and analyzing the data. The results indicate a linear relationship between the bath and cooler temperatures (see Figure 2). The resulting equation can be used to adjust the bath temperature in order to provide a proper cooler temperature for sample storage, although small adjustments of the bath temperature still may be needed. The cooler temperature is recorded with a platinum resistance temperature detector (P-RTD) and a data logger, and will be used as a reference temperature for all soil samples stored in the cooler. The cooler is used so that samples can be placed into the NMR quickly. The measured temperature rise with a soil sample during an NMR measurement was found to be negligible.

Figure 1. Temperature control system (NMR on the left and cooler on the right in the refrigerator, respectively, and temperature bath on the right outside of the refrigerator).

In order to use the NMR analyzer correctly and efficiently, we also determined the soil sample length detected by the NMR by repeating Tice et al.'s experiment (1982). The height of the soil placed in the sample tube was increased incrementally, and the corresponding NMR signal intensity was record (see Figure 3). After 3.5 cm, the NMR signal intensity became constant. Based on these results, soil samples will be prepared with a height of 4 cm to fill the NMR detection zone completely.

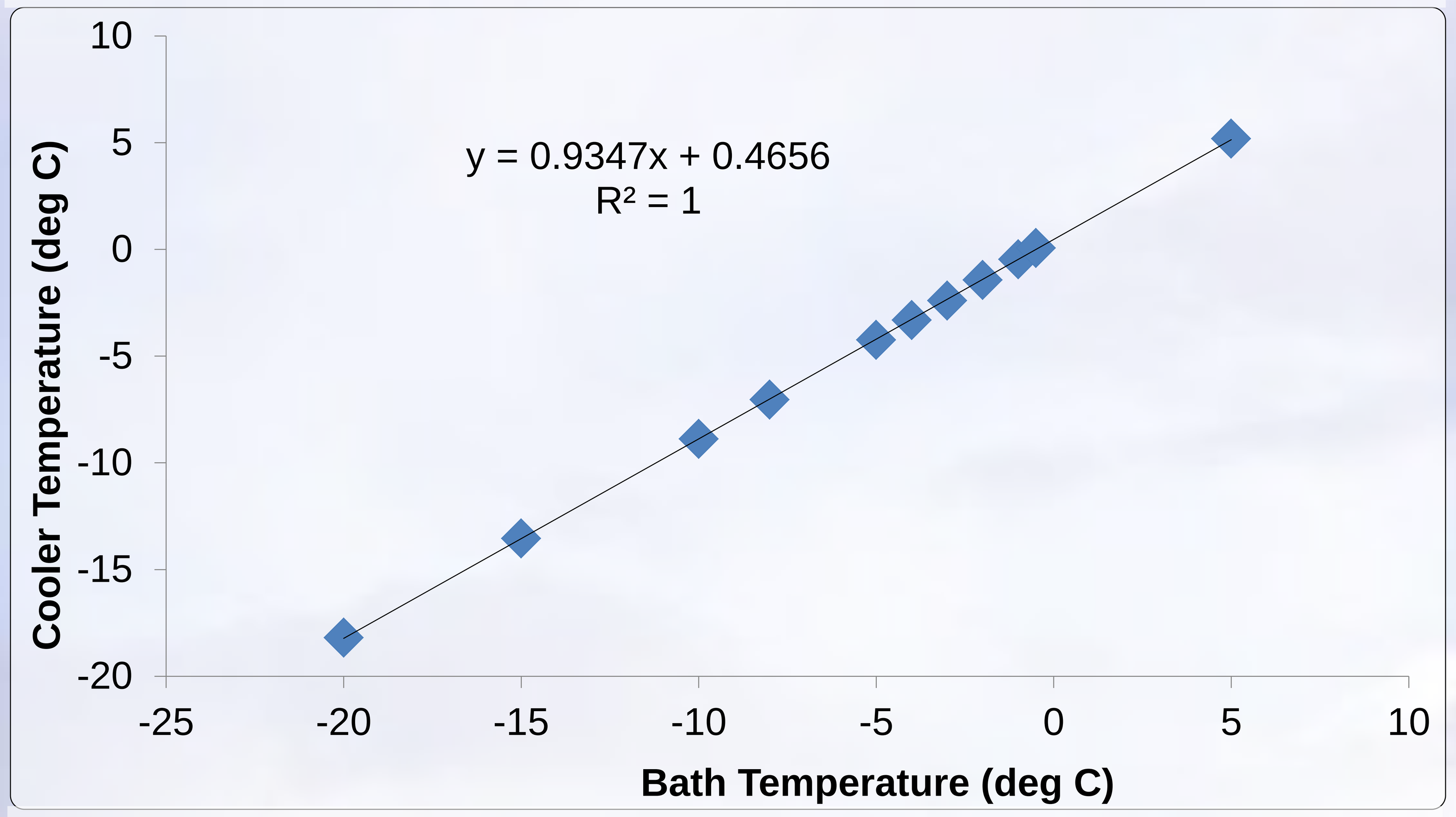

Figure 2. The linear relationship between cooling bath temperature and cooler temperature.

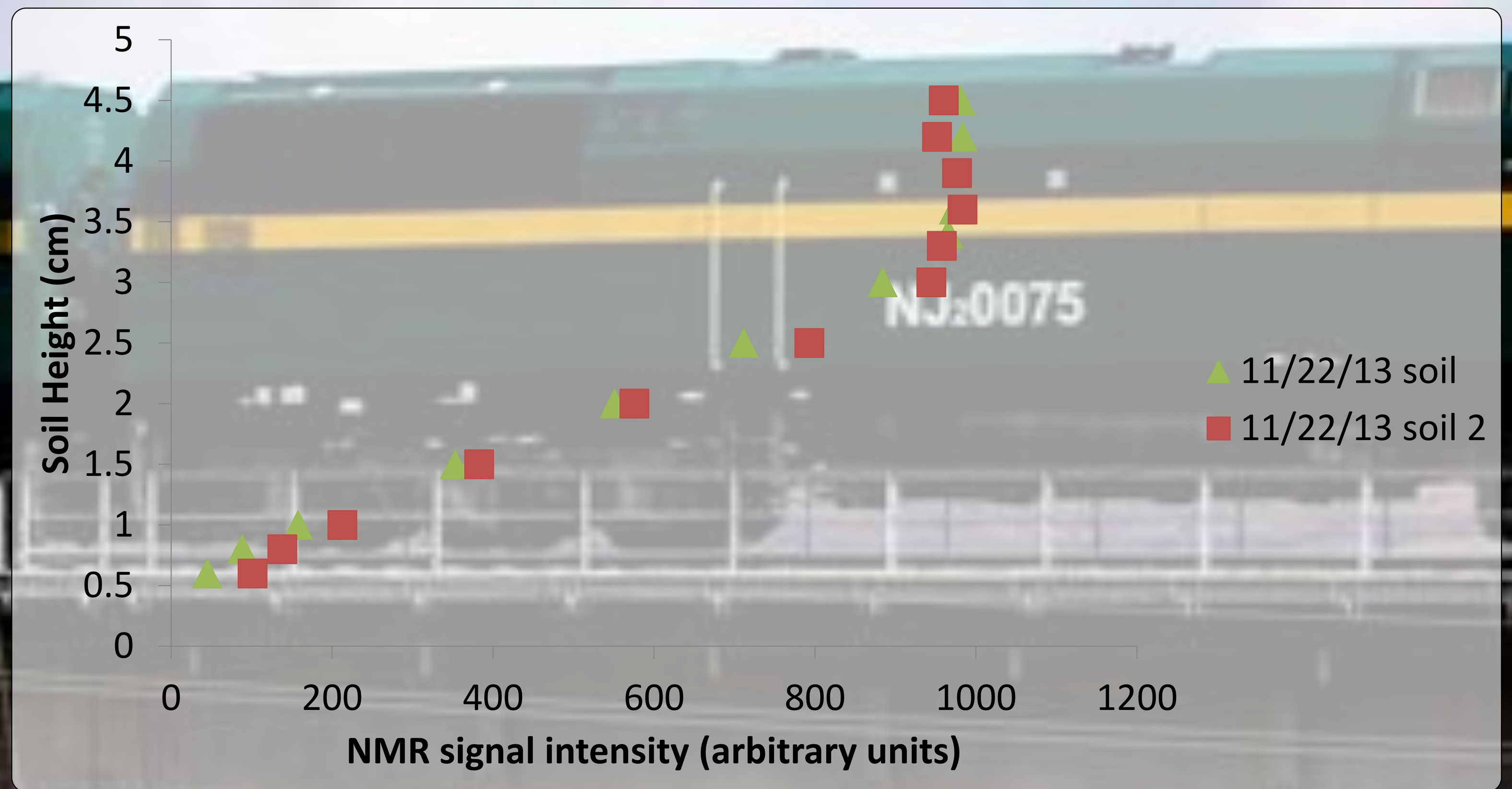

Figure 3. Soil height with corresponding NMR signal intensity.

Results

A test to show that the NMR can detect changes in water content in soil was conducted on silt. Nine duplicate silt samples each with a different moisture content (i.e., 0%, 2%, 5%, 10%, 15%, 20%, 25% 30%, and 40%) were prepared and tested using the NMR at an operating temperature of 34ºC. Figure 4 is a plot of the NMR signal intensity against the sample's moisture content. The results obtained from duplicate samples demonstrate only minor differences, and the relationship between the moisture content of the silt samples and the corresponding NMR signal intensities is linear, thus validating the NMR approach.

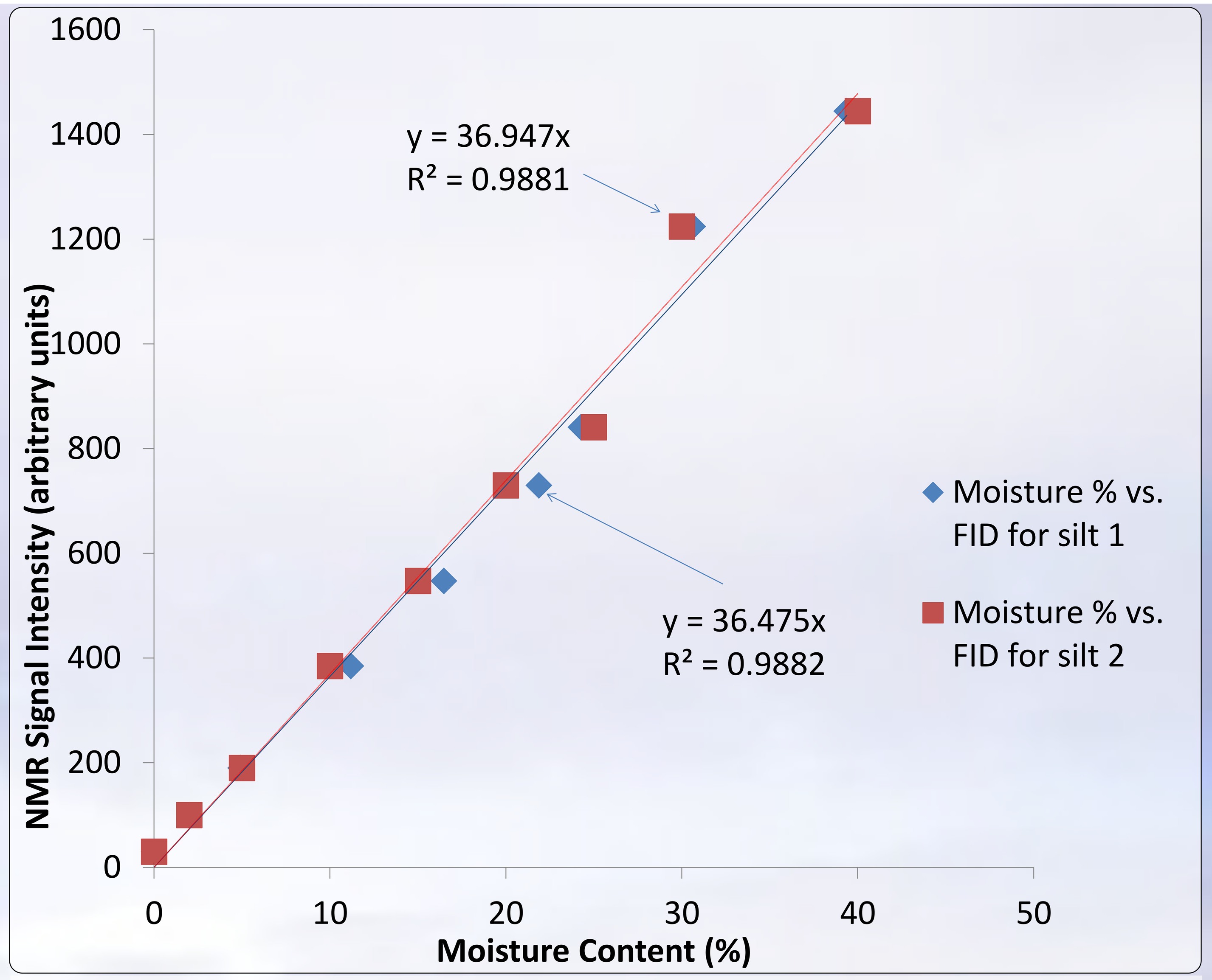

Figure 4. NMR signal intensity for duplicate silt samples with corresponding moisture content.

Conclusion

The approach of using an NMR device to measure the unfrozen moisture content included developing a system to stabilize and control the soil sample's temperature during testing and validating the relationship between the NMR signal intensity and the moisture content of soil sample. Based on these result, we now can proceed with the next phase of this research project, which is to measure the unfrozen water content of cation-exchanged clay samples at below-freezing temperatures, in order to study each soil's ability to retain unfrozen water as a function of temperature.

Reference

Tice, A.R., Oliphant, J.L., Nakano, Y., and Jenkins, T.F. 1982. Relationship between the ice and unfrozen water phases in frozen soil as determined by pulsed nuclear magnetic resonance and physical desorption data. CRREL Report 82-15.

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The background photograph is the Qinghai-Xizang railroad in China, constructed on permafrost, or ground that remains frozen. Results from this research will be applicable to the study of permafrost soils, thermal modeling, and to the design of structures such as this. (Photograph by Henry Chen posted on National Snow & Ice Data Center. https://nsidc.org/cryosphere/frozenground/people.html)