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## Abstract

The overall goal of our research project is to study the unfrozen water mass and mobility in frozen soils. 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. To be successful, our first step was to develop standard procedures for sample preparation. During the past six months, we have developed and tested a set of methods for preparing clay samples, which included crushing source rocks into clay-sized samples with a suitable grain size distribution and exchanging cations for each type of clay. We experimented with different crushing methods, including using a ball mill, and mortar and pestle. Repeatable hydrometer test results indicated that our final combination of methods will produce clay samples with grain size distributions that are acceptable for future testing. Next, we exchanged the adsorbed cations with  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ , and  $\text{Na}^+$  using chloride salt solutions, and flushed the excess chloride from the soil. Each cation-saturated clay required a different number of flushes due to the changes in surface chemistry. Sample preparation may seem simple, but all great research begins with a sound scientific foundation.

## 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. The first step in the research (and the subject of this poster) is preparing five standard clay soils and one heterogeneous soil from Alaska for testing. We acquired five standard clays from the Source Clays Repository: kaolinite and montmorillonite in powdered form; and illite, mixed-layered illite-smectite, and chlorite (i.e., ripidolite) as rock chips.

## Processes and Methods

Sample preparation involved two major processes: crushing rock chips and exchanging surface cations. Each of these processes will be discussed separately.

The first step in analyzing the unfrozen water mass and mobility in frozen soil was to procure fine-grained soil samples. We developed a method for crushing the rock chips (see Figure 1) to the appropriate grain size distributions. It included pre-crushing using a mortar and pestle, passing the sample through a No. 200 (75  $\mu\text{m}$ ) sieve, and finely crushing the portion passing the sieve in a ball mill. Both pre-crushing and sieving involved moistening and drying the samples in an oven. The temperature setting of the oven was critical, as high temperature may affect the clay structure. We chose a temperature setting of 50°C for this research. After the crushing was complete, we conducted specific gravity tests and hydrometer tests to analyze the grain size distribution of each sample. We compared the results with the powdered clay samples and previously-tested heterogeneous soil samples from Alaska, to see if the results were practical. We had concerns about adequately crushing the rock chips to produce a clay-sized fraction in each soil sample.

The soil surface cation exchange involved two stages. First, 40 grams of soil was saturated with a one molar salt solution. The amount of salt required was different depending on the type of salt and its molar mass. For example, the required amounts of sodium chloride (NaCl) and potassium chloride (KCl) were approximately 58.44 grams and 74.54 grams, respectively. To ensure maximum cation saturation, the actual amount of salt used was 1.05 times the equivalent of a one molar solution. Each soil sample and the 1.05M salt solution were divided equally into two one-liter plastic bottles. Both plastic bottles were placed on a shaker table and agitated for two days. The second stage of the soil surface cation exchange was removing the chloride from the soil-brine mixture. A Buchner filter funnel, with a filter paper placed at its bottom for easy soil removal, was used for rinsing the soil samples. The cation-saturated soil was placed in the Buchner filter funnel with approximately 500 mL of deionized water. Both gravity and vacuum were used to rinse the soil through the soil and filter. The funnels were placed over Erlenmeyer flasks, which collected the effluent. The electroconductivity of the effluent was measured using an electroconductivity meter to record the amount of chloride that was removed from the soil. The process of adding 500 mL of deionized water to the soil sample was continued until the electroconductivity was around 10  $\mu\text{S}$  (micro-Siemens). Finally, the rinsed soil was removed from the Buchner filter funnel and placed in a plastic bottle for storage.

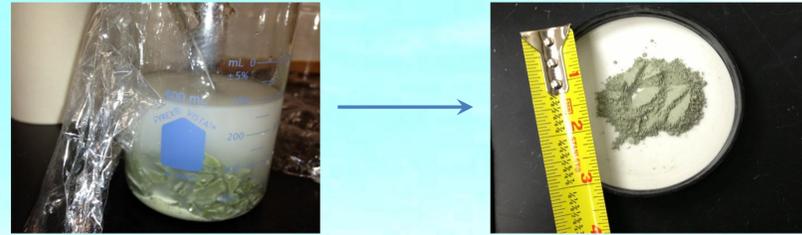


Figure 1. Chlorite (in the form of Ripidolite) before and after crushing.

## Results

The samples have similar specific gravities in the crushed state as shown in Table 1. Figure 2 is a graph of the grain size distribution curves for four of the five clay samples (the remaining sample is currently being processed) and two heterogeneous soil samples that are typical of Alaskan soils (CR is silty clay from the Copper River Lowlands, and FS stands for Fairbanks silt). Duplicate hydrometer tests were run for montmorillonite and kaolinite. The pair of curves for each sample is nearly identical, demonstrating repeatability in the test method. Each of the powdered clays has a much finer grain size distribution, which is a product of the industrial crushing preparation. The size distributions of the original rock chip samples suggest that different rock samples are affected by the samples' physical properties, for instance, their hardness. The similar shapes of the illite and chlorite curves indicate that this combination of crushing methods will produce grain size distributions similar to them. Meanwhile, comparing the curves of illite and chlorite with CR and FS indicates that the samples produced by this crushing method have grain size distributions that are similar to the CR sample, and contain a larger clay-sized fraction. Thus, these prepared samples can be used properly for further testing and analysis.

Table 1. Specific gravity tests for four clay samples.

Sample	Illite	Montmorillonite	Chlorite	Kaolinite
Specific Gravity	2.72	2.71	2.73	2.72

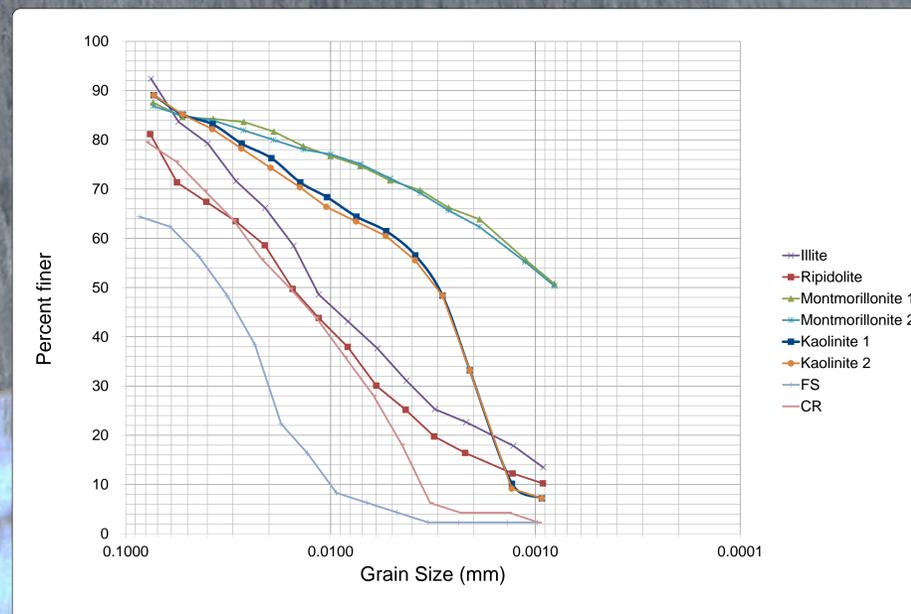


Figure 2. Grain size distributions for four of the five clay samples and two heterogeneous fine-grained Alaskan soils.

Figure 3 is a graph of the electroconductivity versus the number of rinses for montmorillonite. The different electroconductivity values and number of rinses for each of the salts is different for montmorillonite. The results show, in some cases, where a decrease in electroconductivity is followed by an increase. In those cases, the cause is due to the use of vacuum followed by the use of just gravity. When the vacuum is not used, the deionized water is allowed to saturate the soil thereby removing more chloride. The same can be said about the results in Figure 4, which is a graph of the electroconductivity versus the number of rinses for kaolinite. For the montmorillonite saturated with NaCl, this method did not work because the soil sample did not drain. The chloride was removed from this sample using dialysis tubing. For both the kaolinite and montmorillonite, the average electroconductivity reading after rinsing was 8.5  $\mu\text{S}$ , which was less than the targeted 10  $\mu\text{S}$  reading. The electroconductivity results for the remaining soil samples are currently being processed.

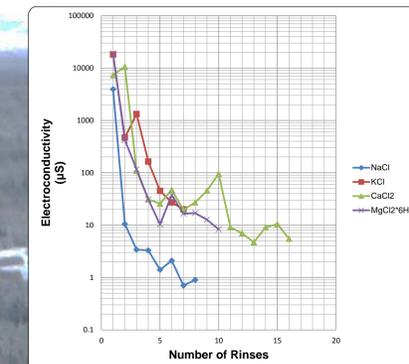


Figure 3. Electroconductivity measurements versus the number of rinses for montmorillonite.

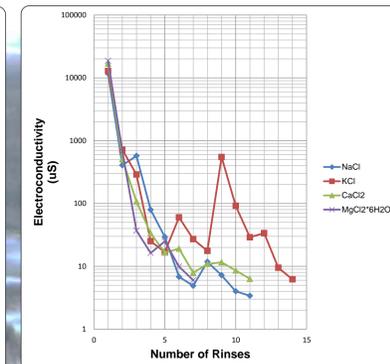


Figure 4. Electroconductivity measurements versus the number of rinses for kaolinite.

## Conclusion

Grain size distribution curves of the soil samples indicate that clay samples in the form of rock chips crushed using these methods will have a practical grain size distribution for further testing and analysis. The cation exchange method and rinsing procedure produced samples that now are dominated by the selected cation and can be used for analyzing cation effects on the surface chemistry for those soil samples. Often not given the credit it is due, developing proper and repeatable methods to prepare samples is necessary for successful research.

## Acknowledgements

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