

**Kearney Foundation Fellowship  
Final Report Summary - Due August 31, 2010**

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**Project Title: Measuring Specific Surface Area of Soils and Soil Minerals by Water Vapor Adsorption**

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**Reporting Period: March 1, 2011- June 30, 2011**

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**Introduction and Research Objectives**

Surface area is related to many physical and chemical properties of soils. Reactive surfaces in soils vary widely because of the differences in mineralogical and organic composition, as well as in particle-size distribution. Some properties closely related to the specific surface area of soils are water retention and movement, cation exchange capacity, heavy metal and pesticide adsorption, and biological processes.

The specific surface area is defined as the surface area per unit mass of soil, expressed in square meters per gram ( $\text{m}^2 \text{g}^{-1}$ ). Specific surface area of soils has been measured in many ways, some are tedious and time intensive. The two common methods are  $\text{N}_2$ -gas and EGME adsorption.  $\text{N}_2$ -gas adsorption requires specialized equipment and is limited to only measuring the external surface area of soil. EGME adsorption measures the total surface area (internal and external). EGME method is only valid on to a single partial pressure equilibration or to an inflection point in a non-equilibration procedure, and samples cannot be used multiple times.

Therefore, the research objectives of this project are to measure the specific surface area of soils and soil minerals quickly and accurately with water vapor, by using a water potentiometer to measure the relative vapor pressure ( $P/P_0$ ) and the weight of adsorbed water vapor. The BET equation will then be applied to determine the amount of adsorbed water molecules at monolayer coverage and calculate surface area from this. Comparisons of surface areas will be made between soil types, clay minerals, saturating cations ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$ ), and EGME surface area.

**Methods**

Three different soils (Grangeville, Holland, and Merced) and two minerals (kaolinite and montmorillonite) will be used to represent a variety and range of soil/mineral properties. Dry soil or clay minerals will be weighed into stainless steel cups (5 cm diameter and by 2 cm tall) in triplicate. The cups will be placed into one of six vacuum chambers where saturated salt solutions control the relative humidity of water vapor in the chambers' atmosphere. The six saturated salt solutions to be used are: NaOH, LiCl, CaBr<sub>2</sub>, K-acetate, CaCl<sub>2</sub>, and LiNO<sub>3</sub>, which create relative humidities of 8%, 11%, 20%, 24%, 32%, and 48% respectively. Each cup will then be weighed periodically for weight gain or loss due to water vapor adsorption and the partial pressure will be measured by a water potentiometer (Decagon WP4C Dewpoint Potentiometer). Knowing the weight due to water adsorption and the partial pressure, the BET equation can be applied to calculate the specific surface area.

Holland and Merced soils were also individually treated with one of four saturating cations ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$ ) by washing with the appropriate cation-chloride salt. The soils were washed with DI water and dialyzed in DI water for several days, changing the water

periodically. The saturated soils were removed from the dialysis tubes, dried, ground, and sieved (2 mm), and were now ready for surface area measurement.

## Results

The results of surface area performed by the two methods are similar except that the water vapor adsorption method gives a slightly higher surface area of soils than the EGME method, but they are comparable (Table 1).

Table 1. Surface area from water vapor adsorption and EGME.

Soil/Mineral Type	Water Vapor Adsorption Surface Area ( $\text{m}^2 \text{g}^{-1}$ )	EGME Surface Area ( $\text{m}^2 \text{g}^{-1}$ )
Kaolinite	8	7
Montmorillonite	348	305
Grangeville	23	16
Merced	163	154
Holland	142	108

Holland and Merced soils saturated with four different cations ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$ ) were expected to have surface area differences due to the hydrated radius of the different saturating cations in the following order:  $\text{Mg}^{2+} > \text{Ca}^{2+} > \text{Na}^+ > \text{K}^+$ . We did not see the differences in surface area following this pattern in either soil (Table 2). However, Na-saturated surface area was greater than K-saturated in both soils. Mg-saturated surface area was greater than Ca-saturated in the Holland soil and about the same in the Merced soil. The divalent cations had a greater surface area measurement compared to the monovalent cations in the Merced soil but the divalent cations had a smaller surface area than the monovalent cations in the Holland soil. The differences in monovalent and divalent cations between the two soils might be due to attraction strengths to the soil surfaces, changing the size of the hydrated radii, and thus the overall surface area measurement. Further experimentation is needed with a variety of cation-saturated soils to determine if strength of cation attraction to the soil surface indeed changes the surface area measurement.

Table 2. Surface area from water vapor adsorption and EGME of cation saturated Merced and Holland soils.

Clay Types	Water Vapor Adsorption Surface Area, $\text{m}^2 \text{g}^{-1}$	EGME Surface Area, $\text{m}^2 \text{g}^{-1}$
$\text{Na}^+$ Merced	108	137
$\text{K}^+$ Merced	94	112
$\text{Mg}^{2+}$ Merced	124	124
$\text{Ca}^{2+}$ Merced	126	130
$\text{Na}^+$ Holland	210	102
$\text{K}^+$ Holland	96	103
$\text{Mg}^{2+}$ Holland	86	92
$\text{Ca}^{2+}$ Holland	57	96

## **Conclusion**

Water adsorption method is a valid method to quickly measure the surface area of soils and soil minerals because the partial pressure can be controlled, the soil weight and the matric potentials are known for every measurement, and the surface area can be calculated using the BET equation. The results of surface area from EGME and water adsorption method are comparable, validating the use of water adsorption to estimate surface area of bulk soils. However, surface area measured after saturating the soil surfaces with different cations were quite different and require further investigation.

## **Describe the major challenges and opportunities or other pertinent information important in the overall achievement of your project.**

The greatest challenge was to prevent the loss of soil particles when saturating the soil surfaces with the different cations. We saturated the soil surfaces by washing the soil three times with the appropriate cation solution. After each wash, some clay-sized particles were most likely poured out, especially if there was soil dispersion. This might explain with differences in surface area between the bulk soil and the saturated cation treated soils. A better method might be to dialyze the soils in a saturating cation solution instead of successive washes.