



A method to determine particle size distribution in soils with gypsum



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ABSTRACT

Gypseous (>40% gypsum) and gypsiferous (1 to 40% gypsum) soils are estimated to comprise from 100 million to 200 million ha globally including over 1 million ha in the U.S. More intensive land use on these soils has resulted in greater demand for information on distribution, properties, and behavior of these soils. Common laboratory methods for determining particle size distribution of soils involve pretreatment to remove gypsum and more soluble salts since they interfere with sample dispersion and establishment of a stable clay suspension. Thus, particle size measurements using standard methods with pretreatment to remove gypsum only reflect the size distribution of essentially insoluble, dominantly silicate minerals, and do not reflect the size distribution of the whole soil including gypsum. Forcing results into a silicate–mineral basis in moderate to high gypsum soils can profoundly distort analytical results. The objective of this project was to develop and evaluate an alternative method of particle size measurement for soils with gypsum that includes size distribution of gypsum particles. Three disaggregation methods for estimation of total sand and sand separates were evaluated: 1) drying sieving, 2) shaking in a 7:3 ethanol:water solution, and 3) sonication in a 7:3 ethanol:water solution. Evaluation of samples with and without gypsum indicated that the sonication method was the most effective for disaggregation as indicated by sand fraction distribution and microscopic grain evaluations. Application of the method to samples from three gypseous pedons yielded grain-size distributions that better agreed with hand textural class estimates than conventional grain-size analysis by standard methods that included pretreatment to remove gypsum. Comparison of CEC/clay and 1500 kPa water/clay ratios for the samples between the two methods also suggested that the proposed method yielded clay contents more consistent with other property data for the same horizons. Improved particle size evaluations for soils containing gypsum, especially those with high gypsum contents, will improve the understanding and ability to interpret and predict behavior of these unique soils.

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1. Introduction

Gypseous (>40% gypsum) and gypsiferous (1 to 40% gypsum) soils are estimated to comprise anywhere from 100 million to 200 million ha globally (Boyadgiev and Veheye, 1996; Eswaran and Zi-Tong, 1991) and over 1 million ha in the U.S. (Lynn et al., 2002). Gypseous and gypsiferous soils in the U.S. occur primarily in arid and semi-arid areas in the western part of the country, and their use has historically been low intensity grazing and wildlife habitat. Consequently, development of a comprehensive inventory of these soils has been a low priority. Increased demand for food, however, has led to the

expansion of grazing and more intensive agriculture on arid and semi-arid lands of the western U.S. where gypseous and gypsiferous soils occur. Additionally, urban and suburban development in this region has increased in recent years, and intensive management and irrigation associated with suburban land use has resulted in greater demand for information on distribution, properties, and behavior of these soils.

Soil Taxonomy (Soil Survey Staff, 2014a) was amended to more comprehensively recognize properties of gypseous soils. Among the revisions was the addition of substitute particle size classes (family level of classification) for soils with $\geq 40\%$ gypsum in the <20 mm size fraction (gypseous soils). The added substitute particle size classes were gypseous-skeletal for gypseous soils with $\geq 35\%$ coarse fragments, and two classes for gypseous soils with <35% coarse fragments; coarse-gypseous ($\geq 50\%$ particles 0.1 to 2.0 mm diameter), and fine-gypseous (<50% particles 0.1 to 2.0 mm diameter). These taxa were established to enable better interpretations of soils with high gypsum, especially those related to water retention and movement. It should be noted

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that these definitions apply to “particles” with any mineralogy including gypsum.

Common laboratory methods for determining particle size distribution of soils, including those containing large amounts of gypsum, involve complete removal of gypsum and more soluble salts since they interfere with sample dispersion and establishment of a stable clay suspension (Gee and Orr, 2012; Porta, 1998; Soil Survey Staff, 2014b). With this approach, particle size measurements only reflect the size distribution of essentially insoluble, dominantly silicate minerals, and do not reflect the size distribution of the whole soil including gypsum (Porta, 1998). Thus, these methods cannot be used to accurately evaluate grain size of gypseous soils for proper placement into the substitute particle size classes and for proper interpretation of gypseous soil physical behavior.

Hesse (1976) and Vieillefon (1979) proposed a method to determine soil particle size with gypsum intact that involves suspension of the sample in a BaCl₂ solution. The underlying theory of this method is that SO₄²⁻ released into the solution from dissolution of small amounts of gypsum in the suspension will combine with solution Ba to form an insoluble coating of BaSO₄ on the gypsum particles, thus preventing further gypsum dissolution during dispersion and sedimentation in aqueous solutions. This procedure may be viable, but its limitations include the potential to alter size of gypsum particles during initial gypsum dissolution and formation of the BaSO₄ particle coatings. Additionally, abrasion of particle coatings during shaking or stirring for dispersion may result in additional gypsum dissolution, which may interfere with dispersion and/or alter the size of the gypsum particles. Laser diffraction has also been used for particle size analysis of gypseous horizons (Aznar et al., 2013). These authors recognized, however, that the method underestimates clay in favor of fine silt.

The objective of this project was to develop and evaluate an alternative method of particle size measurement for soils with gypsum that includes size distribution of gypsum particles.

2. Methods and materials

2.1. Field and laboratory methods

Samples were collected from a Loki pedon (clayey over fine-gypseous, mixed over hypergypsic, thermic Typic Calcigypsid) in Otero County, NM (32° 28.53' N, 106° 23.18' W) and two Pokorny pedons (fine-gypseous, hypergypsic, thermic, shallow Ustic Petrogypsid) in Culberson County, TX (Pokorny 1 – 31° 54.28' N, 104° 21.73' W; Pokorny 2 – 31° 45.85' N, 104° 23.18' W). Sampling was by genetic horizon from a pit wall. (Series names are used for pedon identification only in this paper. The pedons may not meet the series definition. The family classifications of the pedons are correct, however.) One horizon (Byy1) from an additional Pokorny pedon in Culberson County was included in the disaggregation study. Gypsum cemented Byym horizons were identified in the Pokorny pedons. These horizons were weakly to moderately cemented, however, and samples were easily crushed for laboratory analysis.

Samples for laboratory analyses were air-dried and crushed to pass a 2 mm sieve. Analytical methods were those typically used in the USDA-NRCS Kellogg Soil Survey Laboratory and are described in Soil Survey Staff (2014b). Briefly, soil pH was measured for a 1:1 soil:water suspension, and electrical conductivity (EC) was measured on a saturated paste extract. Cation exchange capacity (CEC) was evaluated by NH₄⁺ saturation with 1 M pH 7 ammonium acetate (NH₄OAc). Gypsum percentage was evaluated by dissolution in water, precipitation in acetone, redissolution, and conversion of solution EC to percent gypsum. Calcium carbonate equivalent was measured by acidification and manometric measurement of CO₂ evolution. 1500 kPa water content was evaluated with a pressure plate apparatus.

Texture by feel was evaluated in the laboratory for samples of air dry <2 mm soil material from 20 of the 21 horizons from the three pedons for which particle size was evaluated by the standard and proposed

methods (insufficient sample was available for the Pokorny 1 Byy1 horizon). The textural evaluations were made by two soil scientists with extensive field experience that included experience evaluating horizon texture for soils with high gypsum content. To avoid potential bias, the soil scientists were not given any information related to sample origin or gypsum content before their evaluation.

2.2. Proposed method

The proposed method includes gypsum particles in the analysis scheme and thus, better approximates the grain size distribution of soil horizons than conventional methods that include pretreatment to remove gypsum. Overall, the method consists of two independent measures of particle size of a sample. The first measures the amount and distribution of sand-sized particles including gypsum particles. The second measures clay content of the insoluble, dominantly silicate, fraction of the sample by standard methods which include gypsum and soluble salt removal to enhance clay dispersion. The clay content measured for the gypsum-free separate is re-calculated to a whole soil (including gypsum) basis using the measured gypsum content of the sample (Eq. (1)).

$$\frac{\text{g clay}}{\text{g}<2\text{mm soil}} = \frac{\text{g non-gypsum separate}}{\text{g}<2 \text{ mm soil}} \times \frac{\text{g clay}}{\text{g non-gypsum separate}} \quad (1)$$

where (g clay/g < 2 mm soil) is the calculated value of whole-soil clay content, (g non-gypsum separate/g < 2 mm) soil is the content of non-gypsum residue in the sample expressed as a decimal ([100-% gypsum]/100), and (g clay/g non-gypsum separate) is the clay content of the non-gypsum residue. Most gypsum particles are sand and silt-sized (Al-Barrak and Rowell, 2006; Aznar et al., 2013; Donner and Lynn, 1989; Eswaran and Zi-Tong, 1991; Hesse, 1976; Jackson and Sherman, 1953; Poch et al., 2010). Thus, it is assumed that only minor amounts of clay-sized gypsum particles comprising a small percentage of the sample weight would be removed during pretreatment to remove gypsum and soluble salts. Recalculation of clay concentration from a gypsum-free to whole soil basis should provide a reasonable estimate of total clay in the sample. It should be noted that accuracy of the clay estimate depends on both the accuracy of the measurement of clay in the non-gypsum residue and on the accuracy of measurement of gypsum in the sample. For gypseous soils, measurement of gypsum content may be imprecise since most analytical methods are designed to measure minor components of a sample instead of the major component as would be the case with gypsum measurement in samples with >40% gypsum. Conservative removal of gypsum from gypseous samples is also a challenge since the residual insoluble fraction is a minor component of the sample.

If the whole soil clay content can be reliably estimated, the challenge in measuring particle size distribution of soils with gypsum is to precisely measure the amount of sand, including sand sized gypsum, in the sample and, if desired, the amount in various sand size separates. Silt content would then be estimated by difference.

2.3. Whole soil sand measurement

To measure sand in a whole soil sample containing gypsum, three conditions must be met: 1) weakly-cemented aggregates must be disaggregated to measure size distribution of primary grains, 2) gypsum particles must not dissolve during disaggregation if it is carried out in a liquid medium, and 3) mechanical agitation to aid disaggregation must not fracture primary grains, especially the relatively soft gypsum grains. To meet these conditions, several methods of disaggregation were evaluated including dry sieving with no pretreatment, shaking in a 7:3 ethanol:water solution, and sonication in a 7:3 ethanol:water solution.

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