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Evaluation of the particle sizes of four clay minerals

Xiaoling Tan ^a, Fengyin Liu ^{a,b}, Liming Hu ^c, Allen H. Reed ^d, Yoko Furukawa ^d, Guoping Zhang ^{e,*}

^a Department of Civil & Environmental Engineering, Louisiana State University, Baton Rouge, LA 70803, USA

^b School of Civil Engineering and Architecture, Xi'an University of Technology, Xi'an, Shaanxi 710048, China

^c State Key Laboratory of Hydrosience and Engineering, Tsinghua University, Beijing 100084, China

^d Naval Research Laboratory, Stennis Space Center, MS 39529, USA

^e Department of Civil & Environmental Engineering, University of Massachusetts Amherst, Amherst, MA 01003, USA

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ABSTRACT

The particle size distributions (PSD) of four clay minerals from the US Clay Minerals Society, kaolinite, illite, Ca²⁺-montmorillonite, and Na⁺-montmorillonite, were analyzed using different dispersion techniques to evaluate their PSD variability and intrinsic particle sizes. Both dry dispersion in pressurized air and wet dispersion in water and ethyl alcohol with and without an interlayer expanding agent, glycerol, were employed, along with mechanical ultrasound disaggregation and chemical dispersion. Results show that all four clay minerals exhibit multimodal PSD consisting of superimposed subordinate lognormal distributions from submicron and 1–2 μm primary particles, 10–20 μm flocculi (and trace impurities). Microflocs of 50–500 μm occur only in the dry dispersion mode. Owing to the presence of relatively strong flocculi that are not prone to breakdown to smaller primary particles, the intrinsic PSD of these clay minerals cannot be readily obtained by the adopted dispersion methods. The two swelling clay minerals, Na⁺-montmorillonite and Ca²⁺-montmorillonite, are more sensitive than the non-swelling ones, kaolinite and illite, to different dispersion solvents. The variability of these clay minerals' PSD is further discussed in terms of the complex interactions among clay mineral particles, polar or less polar solvent, and dispersant, such as swelling, cation exchange, exfoliation, and electrical double layer repulsion. A significant practical implication is that most existing dispersion methods for PSD analysis tend to overestimate the silt-sized fraction but underestimate the clay-sized fraction, which may yield misleading classification for clay mineral-rich soils. For expansive clay minerals, appropriate dispersants should be selected to avoid cation exchange between the sample and dispersants, which can change their intrinsic PSD.

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1. Introduction

Clay minerals are the major solid constituents of cohesive soils, most dispersed sediments, and some fine-grained rocks. As a naturally occurring geomaterial, clay minerals are also one of the most important mineral resources used in many industrial and domestic applications. The particle size or size distribution of clay minerals is a key parameter for mineral identification (i.e., usually requiring size separation) and soil's engineering classification, as well as understanding a soil's mechanical, physical, and chemical properties that are in turn of critical importance for many environmental processes and industrial applications, such as water quality monitoring, environmental abatement, sediment transport, oil well drilling, clay-polymer nanocomposites, and nanoclay catalysts (Walling and Moorehead, 1989; Harvey and Murray, 1997; Hill, 1998; Zbik and Smart, 1998; Murray, 1999; Pavlidou and Pappaspyrides, 2008; Anderson et al., 2010; Kiliaris and Pappaspyrides,

2010). For instance, it is a recommended practice that separation and extraction of clay fractions (i.e., <2 μm particles) is performed for qualitative and quantitative analysis, at least for clay minerals (Moore and Reynolds, 1997). In addition, particle size affects a soil's hydraulic conductivity and moisture retention capability (Daoud and Robert, 1992) and dye and clay mineral interactions (Neumann et al., 2002).

However, owing to their micrometer to sub-micrometer sizes, determination and evaluation of clay minerals' particle size distributions (PSD) are not routinely performed in most engineering or industrial practices, usually because of constraints of time or device availability. For example, according to the ASTM Standards (ASTM, 2007), PSD are very important parameters for soil classification, yet no further effort is required to characterize PSD for the <2 μm fraction or to check whether the >2 μm fraction contains non-clay minerals. As a result, all particles in this fraction are collectively treated as clays without further differentiating their sizes (e.g., submicrometer vs. micrometer). On the other hand, some well-crystallized clay minerals (e.g., kaolinite) may have relatively large, >2 μm particles. Although these particles are truly clay minerals having chemically active surfaces, they may be counted

* Corresponding author.

E-mail addresses: zhangg@umass.edu, gzhang@alum.mit.edu (G. Zhang).

toward the silt-sized fraction, which may yield misleading soil classification and lead to inappropriate perception on soils' physico-chemical properties.

The majority of existing techniques for PSD measurements require preparing an ideally or appropriately dispersed clay mineral dispersion, mostly in water. A few issues arise from this sample preparation process. The first one is interactions between clay mineral and water, including the formation of electrical double layer on charged clay mineral surfaces, adsorbed cation hydration, and intercalation of water into expandable clay minerals. Of these interactions, water intercalation in swelling clay minerals can cause an initial increase in particle thickness, which then may result in an increase or decrease in size, depending on the types of interlayer cations or degree of swelling and other environmental conditions, as discussed later; The second one is stability of the dispersion. A non-stable clay mineral dispersion can lead to particle aggregation, resulting in coarser PSD; The third one is the use of chemical dispersants to obtain stable clay mineral dispersions (i.e., no flocculation or aggregation). Owing to clay minerals' cation or anion exchange capability, the cations or anions from dissolved dispersants may change clay minerals' properties and hence alter their PSD. For example, a Ca^{2+} -smectite may be partially or completely converted via cation exchange to a Na^{+} -smectite if a Na^{+} -based dispersant is used. After the conversion, because Na^{+} has a stronger hydration tendency than Ca^{2+} , the Na^{+} -smectite can swell to a higher degree such that its interlayer spacing can theoretically increase to infinity, leading to the formation of exfoliated individual 2:1 layers as separate particles. In contrast, Ca^{2+} -smectite or smectites with other divalent interlayer cations (e.g., Mg^{2+}) have limited swelling due to both the van der Waals and electrostatic attractions between interlayer cations and negatively charged 2:1 layers (Helmy, 1998; Segad et al., 2010). Therefore, swelling may result in either an increase in particle size via water intercalation or decrease by exfoliation.

For clay minerals with variable PSD, another concern arises with the definition and determination of truly representative primary particles. According to the literature, primary particles usually refer to the physically solid, homogeneous, individual particles without aggregation (Zhang et al., 2005). For swelling clay minerals, primary particles are the physically coherent stacks of parallel, equally spaced aluminosilicate layers (Güven, 1992). Therefore, primary particles are intrinsic or inherent to the material, and their sizes should not be affected by different sample preparation techniques. The PSD obtained from a clay mineral dispersion that consists of only primary particles are the intrinsic PSD. That is, the intrinsic PSD reflects the particle sizes of primary particles only, but no aggregates or flocs.

It is worth noting further the importance of appropriate dispersion of dispersed clay mineral particles for PSD analysis. Due to crystal imperfections and isomorphous substitutions, most clay mineral particles possess net negative charges on face surfaces, which are balanced by adsorbed and/or interlayer cations (e.g., Na^{+} , K^{+} , Ca^{2+}). The broken bonds on edge surfaces result in pH dependent charges (e.g., positive if pH is below the isoelectric point). As such, electrostatic forces together with other interactions, such as van der Waals attraction and double layer repulsion, between dispersed clay mineral particles usually lead to aggregation. Therefore, a clay mineral dispersion is a very complex system that contains both individual clay mineral particles and most likely clay mineral aggregates. Together with other unique features of clay mineral particles, such as irregular shapes, flexible layers, and heterogeneous layer charges, clay minerals usually exhibit a wide range of particle sizes even in pure water under stable equilibrium condition (Murray, 2000; Wu, 2001; Lagaly and Ziesmer, 2003).

Both mechanical and chemical methods can be used to facilitate dispersion or disaggregation of clay minerals (Zhang et al., 2005). The former usually involves the application of mechanical energy to breakdown and separate aggregated particles, such as ultrasound disruption, flow-induced shearing in a high-speed stirrer, while the latter refers to the use of dispersants to remove or eliminate the physical

cementation or chemical bonds holding particles together (Zhang et al., 2004, 2005). Therefore, accurate measurement of intrinsic PSD largely depends on the effectiveness of the employed dispersion methods to separate clay mineral aggregates or flocs and keep them stable at their primary particles in a dispersion (Crowley and Welch, 1954). This may also require a continuous dispersion process to maintain the clay mineral dispersion at a completely dispersed, equilibrated state even during measurement. With the development of some new instruments (e.g., laser diffraction-based particle size analyzers), the size of clay mineral particles and their PSD can be quickly measured (de Boer et al., 1987) when a clay mineral dispersion is continuously circulated. Additionally, different dispersion methods, either chemical or mechanical or both, in different solvents (e.g., air, water, or a less polar liquid) can be employed for PSD measurements.

This paper presents an experimental study to evaluate the PSD of four source clay minerals, kaolinite, illite, Na^{+} -montmorillonite (Na^{+} -Mt), and Ca^{2+} -montmorillonite (Ca^{2+} -Mt), which are abundant in natural soils and are representative of four major species of clay minerals with distinctly different behavior. Kaolinite and illite are non-swelling 1:1 and 2:1 minerals, respectively, while the two montmorillonites are swelling 2:1 minerals but with different interlayer cations as well as layer charges. The objectives of this paper are three-fold: (1) to investigate the effect of different dispersion methods on the PSD of different clay minerals, (2) to evaluate the degree of PSD variability of four source clay minerals, and (3) to obtain reference data on the PSD of these representative clay minerals. The findings are expectedly of interest to primarily soil engineers and secondarily researchers who study or use clay minerals for different industrial applications or environmental processes.

2. Experimental

2.1. Materials

Four relatively pure clay minerals, kaolinite (KGa-1b), illite (IMt-1), Ca^{2+} -Mt (STx-1b), and Na^{+} -Mt (SWy-2), which have been well analyzed and widely used by many studies as reference clay minerals, were purchased from the US Clay Minerals Society Source Clay Repository (Hower and Mowatt, 1966; Pruett and Webb, 1993; Chipera and Bish, 2001; Costanzo, 2001). Their geographic origins and basic properties are summarized in Table 1. Kaolinite and two montmorillonites were supplied as dry powder, which had been pre-processed by wet-sieving through a #325 mesh to remove the $>44\ \mu\text{m}$ fractions before shipping to users (Pruett and Webb, 1993). These three "as-received" dry powdery clay minerals were directly used without further treatment. The "as-received" illite sample consisted of small rock chips. To obtain powdery illite, the sample was first hand ground by a mortar and pestle and then wet ground with ethyl alcohol using a McCrone micronizing mill (McCrone Accessories & Components, Westmont, IL, USA) for 3 min, which typically results in a fine powder with $\leq 38\ \mu\text{m}$ particles for most silicate minerals. To identify clay minerals and impurities, the processed powdery illite was side-packed into a sample holder and analyzed by X-ray diffraction (XRD) using a Bruker/Siemens D5000 automated powder diffractometer using a $\text{Cu K}\alpha$ radiation at 40 kV and 30 mA with a scan range of $2\text{--}40^\circ 2\theta$, a scan speed of $0.02^\circ 2\theta$ per 2 s, and a step size of $0.02^\circ 2\theta$. The XRD pattern validates illite as the only clay mineral phase and trace quartz as an impurity (Fig. 1).

2.2. Preparation of clay mineral dispersions

PSD measurements require the preparation of clay mineral dispersions either in air or liquid. Table 2 summarizes the different methods employed to prepare clay mineral dispersions. Details for each method are also provided as follows:

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