Measuring colloidal forces between clay microparticles with optical tweezers

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Abstract

The interaction forces between clay micro-particles play an important role in the macroscopic strength behavior of clayey soils. Optical tweezers were used in the present study to explore the interaction between clay micro- particles. This technology uses a highly focused laser beam to manipulate small objects and can also be used as a force transducer for the measurement of forces on the order of pico-Newtons (pN). Polystyrene beads were first used to measure the surface interactions between polystyrene beads and clay particles for accurate calibration of the system because of their perfectly spherical shape and optical homogeneity, and were successful in obtaining force mea- surements within the range of 20 pN. Subsequently the interactive force was measured when a small clay particle was moved along the surface of a large clay particle. The force measured varies as the interaction of clay surfaces may evolve along their relative motion, leading to force measurements up to 40 80 pN. The present study shows a promising potential of optical tweezers in exploring the complex micro-scale phenomena in clay minerals.

Full Text

Measuring Colloidal Forces Between Clay Microparticles with Optical Tweezers

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Abstract

The interaction forces between clay micro-particles play an important role in the macroscopic strength behavior of clayey soils. In the present study, optical tweezers were used to explore the interactions between clay micro-particles. This technology employs a highly focused laser beam to manipulate small objects and can also function as a force transducer for measuring forces on the order of pico-Newtons (pN). Polystyrene beads were first used to measure surface interactions between the beads and clay particles for accurate system calibration, leveraging their perfectly spherical shape and optical homogeneity. These measurements successfully obtained force data within the range of 20 pN. Subsequently, the interactive force was measured as a small clay particle was moved along the surface of a larger clay particle. The measured forces varied as the interaction between clay surfaces evolved during relative motion, yielding force measurements up to 40–80 pN. The present study demonstrates the promising potential of optical tweezers for exploring complex micro-scale phenomena in clay minerals.

Key words: clay mineral, interaction forces, optical tweezers, montmorillonite

1. Introduction

Clay minerals are primary components of fine-grained soil and rock materials formed through long-term natural weathering processes. These minerals typically occur as clusters or stacks of hydrous aluminium phyllosilicate plate-like sheets with adsorbed water within these layers. Clayey soils and rocks containing significant fractions of clay-size (less than 2 micron) particles are ubiquitous in nature, and understanding their physical and mechanical properties—particularly their resistance to deformation and failure—has profound implications for various engineering applications. These include infrastructure development (e.g., foundations, tunnels, and embankments), hazard assessment (e.g., landslides, debris flows, and earthquakes), and resource exploitation (e.g., mining and water/oil/gas recovery).

Conventional analysis of load and deformation in soils primarily considers skeletal (intergranular) forces transmitted through mechanical contacts between soil particles due to external loading, along with hydrodynamic or seepage forces arising from pore fluid movement through interconnected pore networks [10, 16, 18]. However, due to their small size, complex structural configuration, and affinity for water in the pores, clay particles often exhibit complex behavior influenced by interparticle forces resulting from various physico-chemical processes. These include electrostatic (repulsive or attractive) forces between clay plates, surface and ion hydration forces, electrical double layer forces, and van der Waals forces —largely attributable to the presence of cations and ions in the adsorbed water as well as cations attracted to the often negatively charged clay particle surfaces [10]. The influence of such interparticle forces may not only affect the strength and compressibility properties of soils in traditional engineering analysis but also manifest in complicated scenarios triggered by environmental changes, such as volume changes from expansion and shrinkage during wetting-drying cycles or aggressive contaminant attack.

The critical role of interparticle bonding in clayey soil mechanics has long been recognized [4, 11, 15]. For instance, Morgenstern and Tchalenko [11] demonstrated that shear failure in kaolinite clay occurred between particles rather than within them, indicating that lower interparticle strength governs the overall engineering strength of the soil. Osipov et al. [15] concluded that the nature of interparticle forces could reflect the essence of particle packing and proposed classifying particle assembly patterns based on interparticle distance and force strength to quantify clay structural characteristics. Recent trends arising from increased anthropogenic activities suggest that microscopic interactions among clay particles may become even more significant, as the underlying physical and chemical processes may be enhanced or accelerated. Zhao et al. [23] investigated landslides in China' s Three Gorges area and found that decreased shear strength in soil slopes could be attributed to mineral transformation and structural changes caused by increasing rain acidity. Zhang and McSaveney [22] studied a recent rock avalanche and concluded that air pollution might have accelerated rock strength reduction, consequently triggering rapid sliding of a large rock mass.

Recent efforts to measure and characterize soil particle-level interactive forces have primarily focused on sand-sized particles (typically in the millimeter range). Gao and Hueckel [6] studied polymer growth between two stressed sand grains using Atomic Force Microscopy (AFM), while Michalowski et al. [9] investigated time-dependent deformation growth between two sand particles. The present study aims to explore interactions between clay particles and other mineral particles. Inspired partially by findings of potential clay mineral transformation among three major clay groups—montmorillonite (smectite), illite, and kaolinite—during landslides [23], this study focuses on montmorillonite, a naturally occurring hydrophilic bentonite mineral with a three-layer structure containing an octahedral aluminium layer sandwiched between two tetrahedral silicon oxide layers [5]. Since montmorillonite enrichment is commonly found on landslide sliding surfaces [17, 23], its mechanical properties have attracted considerable attention in landslide hazard assessment [7, 19]. Additionally, due to its large specific surface area, high surface reactivity, and high cation exchange capacity, montmorillonite has been widely used in industrial applications such as catalysis, environmental engineering absorbents, and waste disposal [3].

Various experimental techniques have been developed to directly or indirectly measure forces acting between objects ranging from femto-Newtons (fN) to pico-Newtons (pN) [14]. For instance, Atomic Force Microscopy (AFM) can characterize nanoparticles and provide qualitative and quantitative information on physical properties including size, morphology, and surface roughness [12, 20, 21]. However, for AFM measurements of inter-particle friction, soil particles must be immobilized on a surface and a single particle attached to the AFM tip. The tip-bound particle can then be moved along the surface-bound particle to measure lateral friction. Despite numerous developed methods, lateral AFM force measurements remain difficult to calibrate, necessitating new protocols for these applications. The present study adopts the optical trapping approach as the primary experimental method. The original concept of optical trapping and manipulation of micrometer-sized particles was first reported in 1970 [1] and has since been rapidly developed and broadly applied to diverse small objects including biological molecules, colloidal particles, and living cells [2, 13]. This technology uses a highly focused laser beam to manipulate small objects and can function as a force transducer for real-time measurement of forces on the order of pico-Newtons.

In this study, we employ double optical tweezers to measure forces between montmorillonite particles treated in solution. We aim to develop a technology capable of characterizing interactions between clay particles, which may provide insights useful for understanding the link across scales from microscopic particles to macroscopic clayey soil assemblies.

2. Method

A dry powder sample of montmorillonite was suspended in water and diluted until a suitable particle concentration was achieved. The high concentration of very small particles (a few microns in diameter) makes it difficult to trap only a single particle. To reduce the number of small particles, the sample was quickly centrifuged (20–30 seconds at 800 g) and the supernatant containing small particles was discarded. This procedure was repeated five times.

After this size separation process, the remaining large particles were suspended in a 1:1 mixture of ethanol and pure water. The diluted particle suspension was placed in a measurement chamber consisting of two coverslips sealed with double-sided adhesive tape. Large and small particles with adsorbed water prepared in this manner were then used for the force measurements described below. Only large particles with relatively straight edges were selected to facilitate friction force measurement. The large clay particles used in subsequent experiments were approximately 30 µm in size, while the small clay particles were 2–5 µm in diameter.

It is worth noting that while early optical tweezers development primarily focused on manipulating and moving small objects, the technology has evolved to accurately measure minute forces between small objects, creating opportunities for the present study to measure microscopic forces between clay particles. All experiments were performed on a NanoTrackerTM2 setup with dual beam configuration. The system consists of a laser steering unit and a dual-axis detection unit positioned to independently detect three-dimensional forces and displacements of the two traps [8].

Beam 1 is controlled via a piezo deflection mirror and was used to move small particles along the edges of larger ones. Beam 2 is controlled via AODs (acousto-optic deflectors), a technology that allows generation of multiple traps from a single beam; it was used to hold larger particles in place during measurements. The data presented in this study were recorded in Trap 1 to analyze forces acting on this particle during relative movement. Because the irregular shape and optical inhomogeneity of clay particles constantly cause significant variations in the detected optical trapping signal, a polystyrene (PS) bead was initially used as a force probe for accurate system calibration. Since particle orientation cannot be fully controlled, this 3 µm PS bead was trapped and moved laterally, approximately parallel to the surface of a large clay particle (Fig. 1 [Figure 1: see original paper]). The bead was moved forward and backward multiple times along the same path to find a distance where the bead and clay particle interacted but did not stick completely together, while still generating considerable interaction forces for measurement. The large particle was held at a constant position using multiple traps. Frictional/sliding forces were measured as the trap retracted from the soil particle surface.

During this test, observable interactions between the PS bead and soil particle surface generated stick-slip events, discussed in detail in the next section. This interaction might be further investigated and quantified by approaching the PS bead orthogonally to the clay particle surface. Therefore, a second test measured binding forces between the PS bead and large clay particle (Fig. 2 [Figure 2: see original paper]). The PS bead was moved toward the clay particle surface while measuring binding forces; it was subsequently moved away while measuring changes in these forces.

The bead from previous tests was then replaced with a small clay particle (Fig. 3 [Figure 3: see original paper]) to measure forces between two clay particles. However, accurately controlling the position of clay particles proved difficult; while one clay particle was moved toward another, the timing and nature of their interaction remained largely unknown due to the complex surface conditions of clay particles. This challenge was compounded by the irregular shape and optical inhomogeneity of clay particles, which made optical trapping signal detection much more difficult. In contrast to previous tests using the PS bead, where motion could be fully controlled and the surface was well-defined, enabling reliable force and displacement measurements, numerous attempts were required to approach the small clay particle toward the large one. Eventually, the path shown in Fig. 3 was found to yield stable measurements. Results from all tests are presented in the following section.

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3. Results

In the first test, where the PS bead moved parallel to the soil grain surface (Fig. 1), force data were recorded for the x and y directions in separate channels and are shown in Fig. 4 [Figure 4: see original paper]. The force clearly varied as the bead moved along the surface, though the data cannot distinguish whether the particle was sliding or rotating during movement. The force (Fy) in the y direction appeared unaffected by motion direction. The force (Fx) in the x direction during the first half of backward motion remained almost identical to that in forward motion but then deviated slightly. The jump in the force data (blue curve, at approximately 4.5 μm distance) most likely corresponds to a stick-slip event where the bead temporarily attached to the surface and was released as the optical trap' s pulling force increased. The difference in Fx between forward and backward movement most likely indicates clay particle shape change resulting from the stick-slip event.

The second test explored force evolution during these stick-slip events by orthogonally approaching the PS bead toward the soil particle surface. After brief contact, the bead was retracted and possible binding forces were measured. The results are shown in Fig. 5a [Figure 5: see original paper]. In the retraction curve (red), the bead clearly became stuck to the surface due to attractive interaction between the PS bead and soil particle, coincidentally at approximately 4.5 μm distance. It is instructive to calculate the difference between forces in the approach and retract paths (indicated as "away" and "toward" respectively in Fig. 5a), as shown in Fig. 5b. This difference is very small except during the sticking event. The force change is approximately 20 pN, slightly higher but certainly comparable to the magnitude of change/jump found in the previous test (Fig. 4), suggesting this value could be a reasonable estimate for the PS-clay binding force magnitude.

Fig. 6 [Figure 6: see original paper] shows force measurements as one clay particle moved along a larger clay particle. The measured forces between the two clay particles varied, but periodic peaks were approximately 40 pN, slightly greater than the resultant forces between PS-clay shown in Fig. 4. It is worth noting that clay particles have very complex chemical and physical properties, while PS beads provide a well-defined spherical surface. Multiple sources may contribute to interactions between the two surfaces, including van der Waals forces, hydrogen bonding, and hydrophobic interactions. The measured forces herein may therefore represent a sum of all active interactive forces. Additionally, due to the irregular shape of the large clay particle, the interaction force varied during movement and even increased to around 80 pN at certain positions, which could be attributed to extreme approach between the two particles.

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4. Conclusion

This study explores the use of optical tweezers to investigate complex interactive forces between montmorillonite clay particles. Calibration of optical traps typically requires spherical, optically homogeneous objects; however, soil particles in the samples deviate from this ideal shape, necessitating consideration of larger errors in the calibration procedure for force measurements.

Attempts to use perfectly spherical polystyrene beads to measure surface interactions between PS beads and soil particles successfully obtained reasonable force measurements within the 20 pN range. Subsequently, measurements of binding force evolution between two montmorillonite microparticles yielded values around 40 pN. It should be noted that the optical inhomogeneity of the soil particles would cause detection signals during rotation, making accurate measurements very difficult to achieve. Future studies may consider developing methods to couple soil particles to spherical beads (silica or polystyrene) and to immobilize large particles on a glass surface. This would reduce potential errors from unwanted particle movements and rotations. The present study has demonstrated the promising potential of optical tweezers for better understanding complex micro-scale phenomena in clay minerals.

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