

Particle size distribution of kaolin clay with phase-contrast nano-CT

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1 Introduction

The particle size distribution (PSD) of granular materials plays a pivotal role in understanding the physical and mechanical behaviour of clays in civil engineering applications. Accurate determination of PSD is essential to optimise the mechanical, thermal, and electrical properties of fine-grained materials. Furthermore, in absence of more detailed laboratory tests, the PSD is instrumental in engineering practice for ground improvement methods, analyses of susceptibility of frost heave, and selecting TBM configurations (face stability).

Traditional approaches to PSD determination, such as sedimentation techniques, dynamic light scattering (DLS), static light scattering (SLS), and laser diffraction, remain the backbone of geotechnical and colloidal science practice. However, these techniques face limitations: they rely on simplifying assumptions regarding particle shape, density, and dispersion state. In particular, they cannot easily distinguish between individual clay platelets and aggregates (flocs), which can result in systematic errors in PSD determination.

This study investigates the use of phase-contrast nano-X-ray Computed Tomography (nano-CT) as an innovative tool for characterising the PSD of clay, comparing its capabilities with DLS and hydrometer analysis. The aim is not to propose nano-CT as a replacement for established techniques in engineering practice, but rather to highlight the insights it provides into the limitations of current PSD measurement methods.

Nano-CT introduces a paradigm shift in clay characterisation, offering 3D, non-destructive imaging with high spatial resolution. For the first time, individual clay platelets can be observed in their natural, undisturbed state, offering a new perspective on the sources of error in conventional approaches.

2 Material

The study was conducted on Speswhite kaolin clay, a highly refined kaolin clay with a relatively uniform granulometry (median particle diameter $D_{50}=1.3 \mu\text{m}$) produced by IMERYS, France. The material was selected because, while remaining a naturally occurring material, it is widely regarded as a model clay for its monomineral monogranular characteristics.

For all methods investigated, a dilute suspension was prepared at different clay concentration (depending on the technique) in demineralised, de-aired water. Only the Speswhite kaolin powder form #200 sieve (passing fraction 0.075 mm) was employed for the clay-water solutions.

3 Methods

3.1 Phase-contrast nano-CT

This study employs a subvolume ($25 \times 25 \times 25 \mu\text{m}^3$ at 50 nm/px) of a 3D tomographic scan of Speswhite kaolin clay suspended in acidic water. The measurement was obtained at the ID16B beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble.

A mixture of clay and acidic water at 10 g/L was pipetted into a quartz capillary tube of 300 μm diameter. The scan was taken after an initial sedimentation phase where the larger clay aggregates settled at the bottom of the capillary tube. More details on the experimental setup can be found in Örtendahl et al. (2025).

The imaging technique used in ID16B is phase-contrast in-line holo-tomography (Cloetens et al., 1999), which enables the submicron spatial resolution and phase-contrast imaging to discern individual clay particles and aggregates from the suspension fluid.

By leveraging AI-based methods for particle segmentation and labelling (cIAI tool developed at Chalmers University of technology, see Örtendahl et al. (2025)), it was possible to quantitatively analyse the obtained 3D-image and accurately identify and quantify the individual particle volumes and shapes in suspension. Only individual platelets (no flocs or aggregates) were used in the determination of the PSD for comparison with the other techniques in which particle separation was promoted.

3.2 Dynamic Light Scattering

The PSD was also determined by Dynamic Light Scattering (DLS) technique using a Malvern ZetaNano ZS available at Chalmers University of Technology. DLS determines particle sizes (between 2nm and 2mm) by analysing fluctuations

in scattered light intensity caused by Brownian motion, with the Stokes–Einstein equation converting these into hydrodynamic diameters.

A clay-milliQ water suspension (0.25 g/L, 10 mL) was pipetted into a 1x1 cm² quartz cuvette and sonicated for 15 min to minimize aggregation before testing.

3.3 Hydrometer analysis

To determine the PSD via hydrometer analysis, a solution of 50g/L of kaolin powder in demineralised water and dispersant agent (to enhance particle separation) was let sediment in a 1L cylindrical container. The sedimentation tests were performed via hydrometer analysis according to ASTM D422-63.

4 Results

The PSD curves from the three methods are shown in Fig. 1.

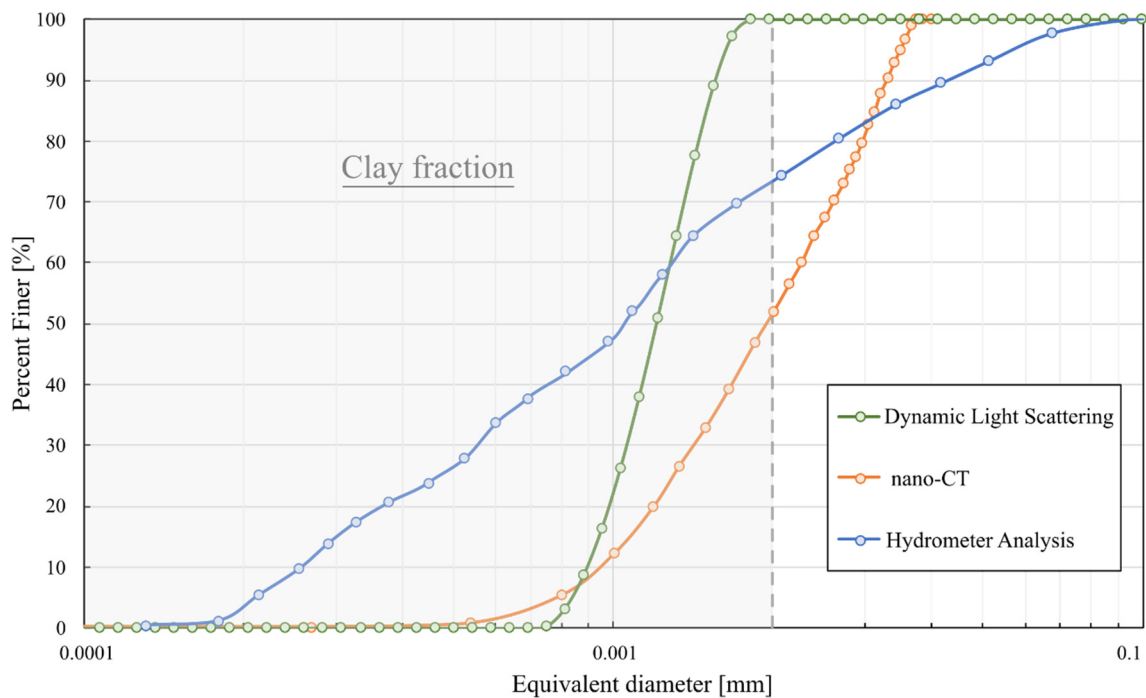


Fig.1: Comparison of particle size distributions obtained from Nano-CT, hydrometer analysis, and DLS.

Nano-CT enables direct 3D observation of discrete clay platelets and is therefore used as ground truth in this study, despite its resolution limit of $\sim 0.3 \mu\text{m}$ resulting from the pixel size of the scan. While the method captures the non-spherical morphology of particles, segmented volumes are expressed as equivalent diameters for comparison (Fig.1). The median particle size obtained is $D_{50, \text{nanoCT}} = 2.0 \mu\text{m}$.

Hydrometer analysis provides reliable size distributions from the #200 sieve (0.075 mm) down to ~ 0.001 mm. However, density measurement bias and the

assumption of spherical particles in Stokes' law lead to overestimation of the fine fraction relative to Nano-CT and DLS (Figure 1), consistent with Chaney et al. (2000). The method yields $D_{50,hydr}=1.0 \mu\text{m}$.

DLS shows a narrower distribution with a median particle diameter of $D_{50,DLS}= 1.2 \mu\text{m}$ (Fig. 1). The technique seems to under detect larger particles. Especially in polydisperse sample (a mix of different particle sizes) with non-spherical particles, their shape, orientation, and rotational diffusion affect their Brownian motion, and therefore, can bias the hydrodynamic diameter toward smaller values.

5 Conclusions

The results of the study reveal that while conventional methods remain indispensable for quick and routine analyses, they often fall short in accuracy of the characterisation of the PSD of clay. For the first time, nano-XCT provides direct 3D visualisation of individual clay particles in suspension, making it possible to critically assess the assumptions underpinning standard methods.

The findings underscore that:

- Hydrometer analysis systematically overestimates the fine fraction due to density bias and spherical particle assumptions;
- DLS underrepresents larger, non-spherical particles, leading to narrower distributions;
- Nano-CT offers direct 3D insight into particle morphology and size but is limited by resolution and not suited for routine practice. While it is not intended as a practical tool for geotechnical engineering, its contribution lies in deepening our understanding of clay behaviour and guiding the more informed use of conventional PSD techniques.

6 References

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