Rheo-optical Analysis of Natural Sediments Under Continuous and Oscillating Shearing Action (EP31E-2406)

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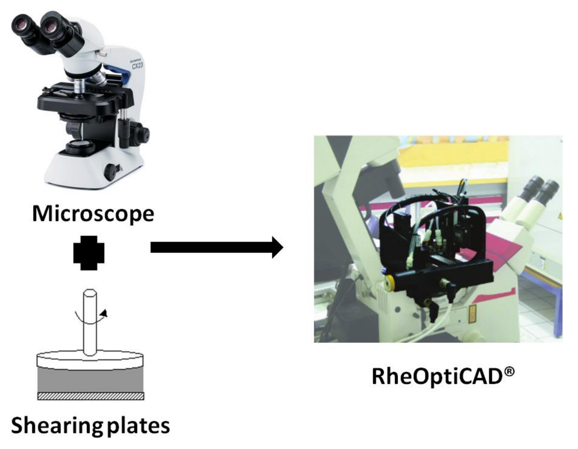
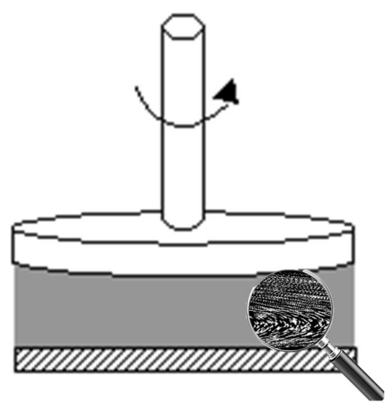
Abstract

A modified version of the commercially available RheOptiCAD® was developed to examine the conformational changes and break-up of flocs in clay suspensions in microscopic detail, under shearing action. In contrast to the RheOptiCAD®, our device aims to enable the structural observation of suspensions even when coagulation or settling of particles onto the bottom plate of the shear cell occurs. This challenge was met by using an upright modular microscope instead of an inverted one, equipped with a CMOS camera to record the structural evolution of samples as a function of shear. The verification of the design was done by analysing a model system of un-flocculated and flocculated kaolin suspensions. Additional experiments with natural clay (mud) suspensions were performed.

Introduction

Optical rheometry, also known as rheo-optics, is a powerful technique to analyse the behaviour of these complex systems, as it allows the visualization of flow, deformation and restructuration of the system under shear.





Many rheo-optical devices developed so far are laboratory models and only some of them have been commercialized.[1-3] Recently, a novel parallel plate rheological device (RheOptiCAD®) was designed by CAD Instruments and reported by Boitte et al.[4] The device has primarily been designed to be mounted on an inverted microscope. The device is however not suited to analyse the optical behaviour of suspensions containing sedimentating particles under shear as this would lead to the blockage of the field of view of the inverted microscope. This led us to propose an alternative set-up, making use of an upright microscope.

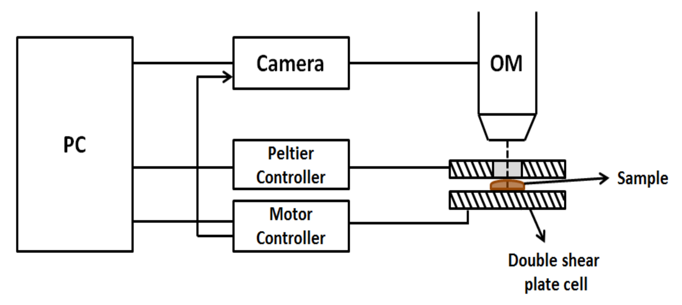


Figure 1. Schematic representation of complete software configuration

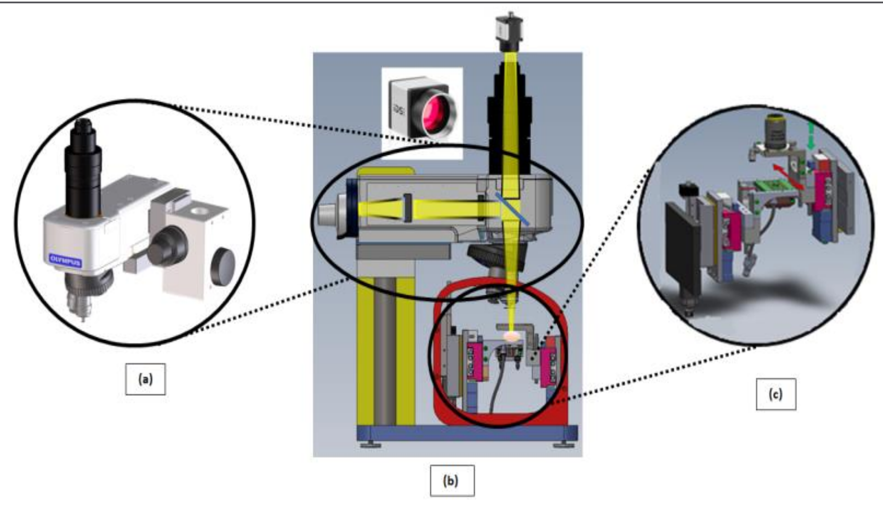


Figure 3. (a) The Olympus microscope, (b) The optical layout of device, (c) Shear movement of the plates

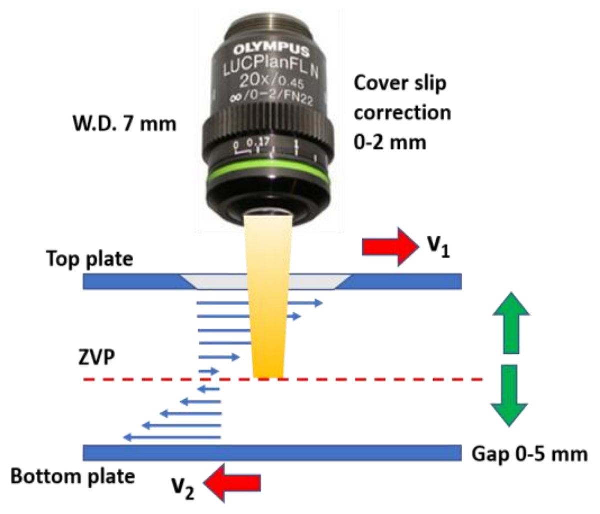


Figure 2. Movement of top and bottom plates along x-axis to change the location of ZVP

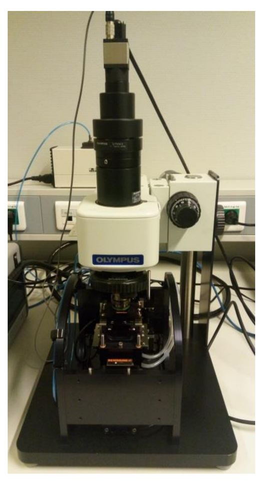


Figure 4. Shear cell combined with an upright optical microscope and a camera

Materials and Methods

The validation of the new set-up was performed with kaolin suspensions in water. Un-flocculated kaolin suspensions were prepared by dispersing small amount of kaolin (Imerys, England) in distilled water. Two commercial polyelectrolytes, were used to prepare flocculated kaolin suspensions, by simply dispersing small amounts of polyelectrolytes and kaolin in distilled water. A natural mud sample, collected from Port of Hamburg (Germany), was also chosen for the investigation. The samples were placed on the bottom plate of the device and a gap of 10-100 µm was set to perform the experiments at 20°C. Several investigations were performed in oscillation mode using frequencies *f* between 0.5 and 2 Hz and amplitudes *A* ranging from 0.1 to 0.5 mm for the bottom plate.

Results and Discussion

Figure 5 shows the snapshot from the video recording for the un-flocculated kaolin suspension. It can be easily seen from the image that the unmodified kaolin particles are very small and homogeneously dispersed within the water. Under oscillatory shear, the particles showed a little bit movement due to the absence of any interactions between the particles.

Figures 6 and 7 present the images of the kaolin suspensions containing polyelectrolytes. Fig. 6 displays the break-up of a flocculated structure by the application of a oscillatory shear for kaolin particles coated with cationic polyelectrolyte. Fig. 7b shows the stretching of a flocculated structure made of kaolin and anionic polyelectrolyte.

The microscopic images of the natural mud sediments are displayed in Figs 8 and 9. Fig. 8b shows the breakage/separation of a bigger flocculated structure into two flocs which further divided into more smaller flocs as shown in Fig. 8c. Fig. 9a shows the presence of a large particle (clay) in the suspension, which displayed the rotational motion during oscillatory shearing as evident in Fig. 9b. This large particle also creates a void during oscillation after 10 s, as shown in Fig. 9c.

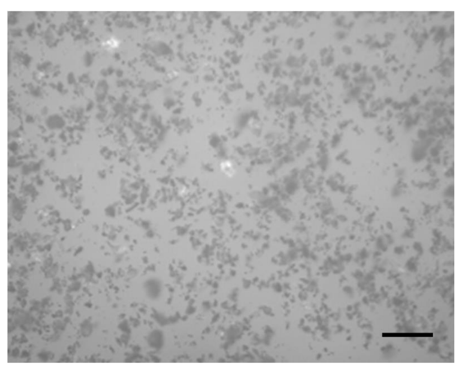


Figure 5. Image of un-flocculated kaolin suspension subjected to oscillation

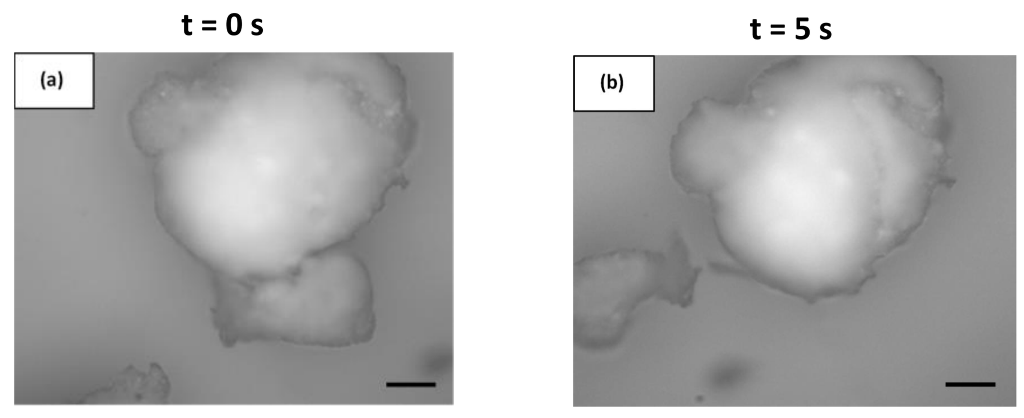


Figure 6. Images of cationic polyelectrolyte-based kaolin subjected to oscillation at (a) t = 0 s (b) t = 5 s; Scale bar represents 70 µm

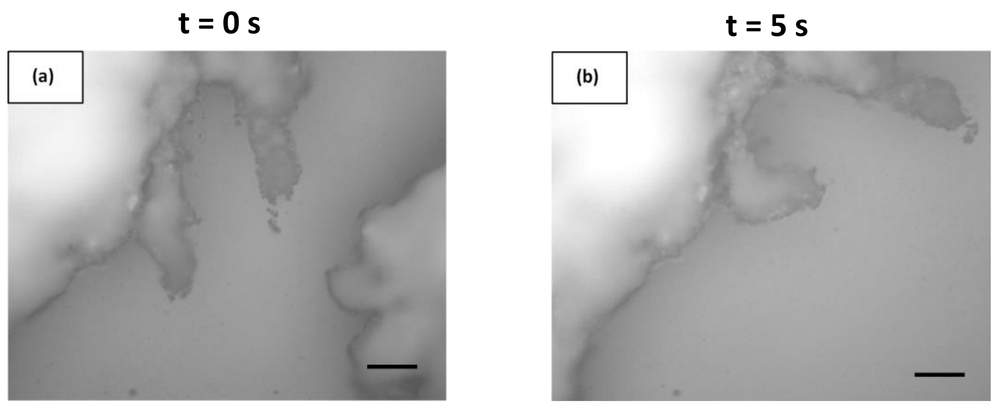


Figure 7. Images of anionic polyelectrolyte-based kaolin subjected to oscillation at (a) t = 0 s (b) t = 5 s; Scale bar represents 70 µm

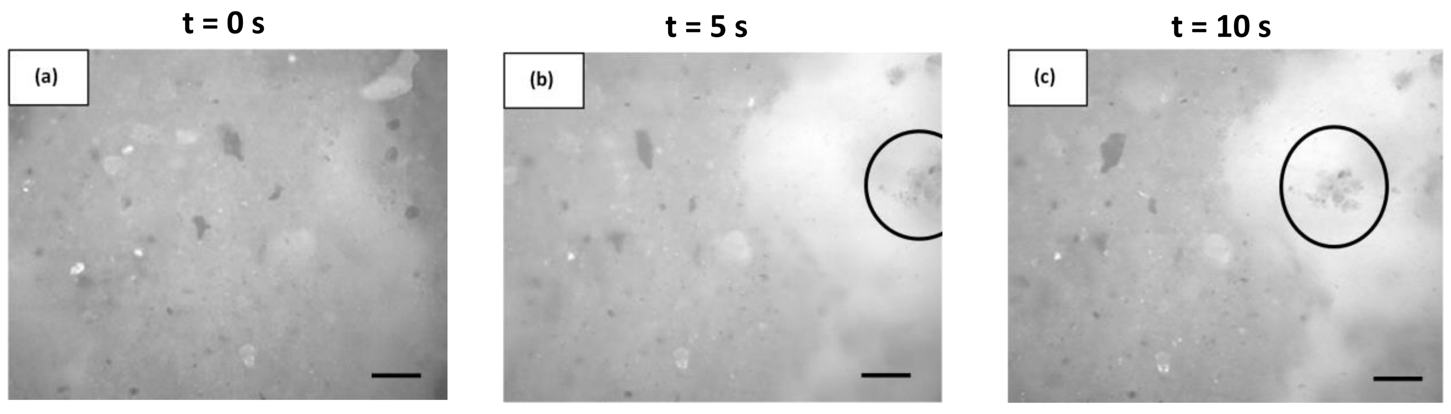


Figure 8. Images of natural sediments subjected to continuous strain at (a) t = 0 s, (b) t = 5 s, (c) t = 10 s. The black circle shows a small floc that has detached from a bigger one on the right. Scale bar represents 70 µm

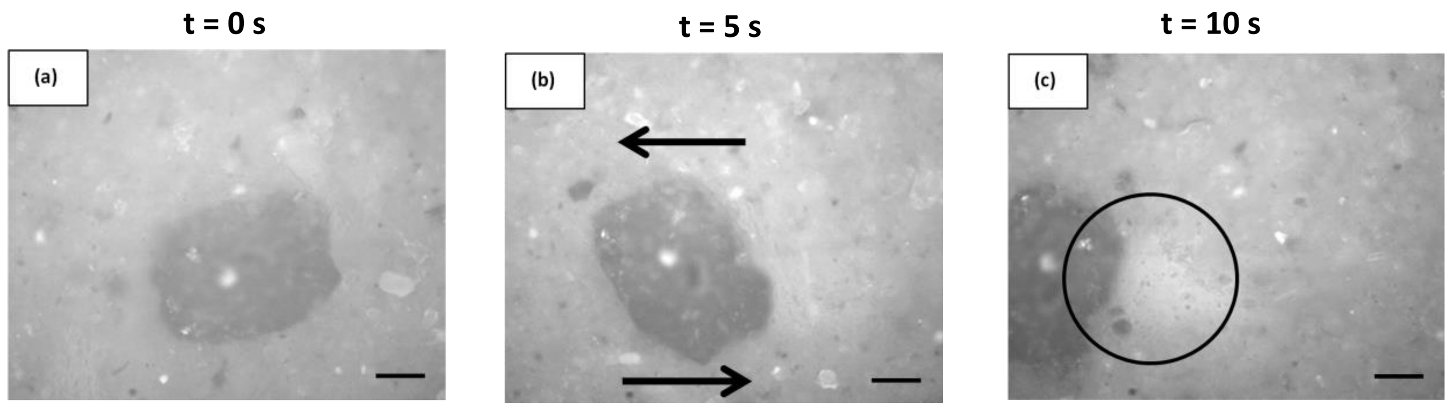


Figure 9. Images of natural sediments subjected to oscillation at (a) t = 0 s, (b) t = 5 s, (c) t = 10 s. Arrows shows the direction of rotational motion of particle. The black circle represents a void Scale bar represents 70 µm

Conclusions

This study presents the modification of an already reported rheo-optical device, which enables the observation of sedimentating suspensions, by using an upright optical microscope configuration.

Proof-of-concept experiments confirmed the applicability of our device for investigating complex systems. Successive snapshots taken from the video recording of these suspensions under shear revealed the structural changes of these systems as a function of the shearing action.

The new device will be used, in the future, to perform state of the art research in the field of sediment rheology by linking the qualitative structural break-up and build-up (thixotropy) of suspensions observed by rheo-optics to the quantitative rheological measurements obtained from conventional rheometer.

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