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1                   **Design of a Parallel Plate Shearing Device for Visualization of**  
2   **Concentrated Suspensions**

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30 **Abstract**

31 A modified version of the commercially available RheOptiCAD® was developed to visualize the  
32 microscopic structural changes occurring in concentrated suspensions, such as the break-up of flocs in  
33 clay suspensions, under shearing action. This is made possible by replacing the inverted microscope  
34 used in the traditional RheOptiCAD set-up by an upright modular microscope equipped with a CMOS  
35 camera and epi-illumination. Our device retains the following features of the previous version of  
36 RheOptiCAD®: [i] uniaxial translational motion of two parallel plates, [ii] three modes of shear  
37 straining, [iii] controlled thermal environment, [iv] vacuum joining of microscopy glass slides. The  
38 validation of the new design was done using a model system of un-flocculated and flocculated kaolin  
39 suspensions and concentrated natural mud suspension. The results showed that the constructed device  
40 is a promising tool for studying, from fundamental and industrial perspectives, the microstructural  
41 behaviour of complex suspended systems under controlled thermal and mechanical conditions.

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50 **Keywords:** Shearing cell, Strain-controlled, Optical microscope, Rheo-optics, Suspensions

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## 52        **1. Introduction**

53        Complex systems, such as emulsions, colloidal suspensions, gels, polymeric and surfactant  
54        solutions, foams and pastes are commonly part of food and non-food products, consumed on  
55        daily basis. For these products, the study of their viscoelastic properties is key for industrial  
56        purposes. Shearing action combined with parameters like pressure, temperature, ageing time,  
57        ionic strength or pH can lead to structural changes in such complex systems. One of these  
58        changes is demixing or phase separation, driven by either gravitational gradient or  
59        thermodynamic forces. Optical rheometry, also known as rheo-optics, is a powerful technique  
60        to analyse the behaviour of these complex systems, as it allows the visualization of flow,  
61        deformation and restructuration of the system under shear. Combining standard rheological  
62        measurements with rheo-optics provides an understanding of rheological parameters such as  
63        yield strength, viscosity or thixotropy in relation with the observed structural changes.

64        With the progress in advanced microscopic techniques, many research groups have developed  
65        devices that combine microscopy and rheology [1-4]. The progress in optical shearing devices  
66        until 1998 has been summarized by Fuller [5] and Wagner [6] for 2D rheo-optics and 3D  
67        rheo-optics, respectively. Later, van der Linden et al. [7] presented the review of rheo-optical  
68        devices for food and non-food systems. The rheo-optic devices developed so far are based on  
69        either an optical device fitted to the commercial or existing shearing device, or alternatively a  
70        shearing system developed to combine with a commercial or existing optical device. The  
71        established accuracy of commercial rheometers led researchers to favour the development of  
72        optical techniques fitted to the existing rheometers [8]. However, these devices have some  
73        drawbacks, *e.g.* limited field-of-view and low magnification power. Custom-made shearing  
74        cells are very interesting alternatives due to the fine tuning and huge flexibility in the  
75        materials selection for the surfaces which come into contact with the sample, the possibility to  
76        analyse samples under larger deformations and the possibility to create a zero-velocity plane

77 (ZVP) at any position within the cell gap by having top and bottom plates moving in opposite  
 78 directions [9, 10].

79 Several optical techniques have been used in rheo-optical devices, depending on the material  
 80 under investigation and the observation scale. Different shearing devices like a 4-roll mill  
 81 [11], controlled strain rheometer [12], parallel plate [13, 14], and others [15] have been used  
 82 so far to perform deformation under controlled environment. Table 1 summarizes some  
 83 important details about the already reported rheo-optical systems, in comparison to our  
 84 designed system.

85 Table 1: Key characteristics of some of the already reported rheo-optical systems

Rheometer/Shearing Cell	Microscope/Camera	Advantages	Limitations	Ref.
IR-200 Rheometer without transducer, Quartz Cone & Plate/Plate-plate geometry	Upright Optical microscope, CCD camera, halogen white lamp	Rotation can be reversed, mechanism for temperature control	Largest image size 640 x 480 pixels, not suitable at high shear rates	[16]
Custom made parallel plate shear cell	Inverted confocal scanning laser microscope	Higher accuracy from mechanical point of view	Lengthy process for sample preparation, requirement of pair of cassettes for every analysis, difficulty in reproducibility, absence of temperature control mechanism	[17]
1. Custom made parallel plate shear cell 2. Anton-Paar MCR 301 Rheometer, Cone & Plate/Plate-plate geometry	1. Upright microscope objective with CCD camera, LED backlight source 2. Inverted microscope objective with CCD camera	1. Allows to shear a very large surface of sample ( $\sim 70 \text{ cm}^2$ ) 2. Measurement of stress	1. Control of gap variation between the plates was difficult, stress measurement was not possible, possible movement of only bottom plate, no mechanism for temperature control 2. Not suitable for suspensions due to inverted microscope	[14]
Custom made parallel plate shear cell	Confocal scanning laser microscope, CCD camera	Temperature controlling mechanism	Fixed gap, movement of only one plate	[18]
Custom made cone & plate shear cell	Inverted confocal scanning laser microscope, He-Ne laser	Movement of both plates, imaging of planes parallel as well as perpendicular to glass plate	Fixed position of microscope objective in cell, variation in gap width caused by a slight wobbling of the glass plate, not suitable for higher shear rates, no temperature controlling mechanism	[9]
Stress-controlled Bohlin Gemini Rheometer, Cone & Plate geometry	Inverted confocal scanning laser microscope	A wide range of shear strain, up to 50% amplitude	Fixed position of microscope objective in cell, no recording of images (absence of camera)	[19]
Linear parallel-plate shear cell	Inverted fast confocal microscope	Wall slip prevention by coating the slides with disordered layers of colloid, solvent trap to minimize evaporation	Possible movement of only top plate, no temperature controlling mechanism	[2, 20]
Custom made parallel plate shear cell	Inverted fast-scanning confocal microscope	Teflon sheets to minimize evaporation, synchronization of image acquisition and shearing	Possible movement of only bottom plate, image size 512 x 512 pixels	[21]

		action		
Custom made parallel plate shear cell	Inverted microscope, CCD camera	Roughened plates to minimize wall slip, movement of both plates	Image size 1024 x 1024 pixels, only oscillatory mode of shearing	[22-24]
Anton-Paar MCR 301 Rheometer, Cone & Plate geometry	Inverted confocal scanning laser microscope	Sandblasted plates to minimize wall slip, solvent trap to minimize evaporation	Image size 512 x 512 pixels, fixed gap width between cone and plate	[25]
Velocity-controlled Couette rheometer	Inverted fluorescent microscope, CCD camera	Allows to access local velocities up to 1 m/s	Not suitable for suspensions due to inverted microscope	[3]
Stress-controlled rheometer AR2000, Cone & Plate/Plate-plate geometry	Inverted confocal scanning laser microscope	Roughened plates to minimize wall slip, solvent trap to minimize evaporation	Not suitable for suspensions due to inverted microscope	[8, 26]
Custom made parallel plate shear cell	Inverted confocal scanning laser microscope	Temperature controlling mechanism, vacuum joining of glass coverslip with the plate, movement of both plates	No stress measurement, not suitable for suspensions due to inverted microscope	[10, 27]
Custom built constant stress shear cell, Cone & Plate geometry	Inverted fast-scanning confocal microscope	Solvent trap to minimize evaporation, cost-effective, availability of range and resolution of applied stresses through selection of transfer fluid	Absence of temperature controlling mechanism, not suitable for suspensions due to inverted microscope	[28]
Custom made parallel plate shear cell	Inverted confocal scanning laser microscope	Stress measurement, Solvent trap to minimize evaporation, possibility of biaxial shear experiments	Absence of temperature controlling mechanism, gluing of cover slip with the plate, not suitable for suspensions due to inverted microscope	[4]
Anton-Paar MCR 301 Rheometer, Cone & Plate/Plate-plate geometry	Inverted laser scanning confocal microscope	Wide range of applied torque, presence of normal force sensor	Not suitable for suspensions due to inverted microscope, image size 256 x 256 pixels	[29]
Rotational rheometer HAAKE MARS III, Cone & Plate/Plate-plate geometry	Inverted polarized reflected light microscope, CCD camera	Temperature controlling mechanism, in addition to light microscopy RAMAN spectroscopy measurements also available	Not suitable for suspensions due to inverted microscope setup	[30]
Custom made parallel plate shear cell	Upright optical microscope, CCD camera	Temperature controlling mechanism	Possible movement of only bottom plate, suitable for small amplitude deformation	[31-33]
Custom made parallel plate shear cell	Upright optical microscope, CMOS camera, LED light	Suitable for suspensions due to upright microscope, temperature controlling mechanism, vacuum joining of glass coverslip with the plate, movement of both plates, gap variation between two plates is possible from 0-5 mm, CMOS camera instead of CCD which provides reduced blooming and smearing, image size 2592 x 2048 pixels with square pixels for undistorted image, fluorescent marker is not required, microscope objective with large working distance	No mechanism for stress measurement	This study

86

87 Many rheo-optical devices developed so far are laboratory models and only some of them  
88 have been commercialized. The Cambridge Shearing System [31] produced by Linkam  
89 Scientific Instrument Ltd. is the first commercialized system for optical analysis under  
90 rotational shearing action. A confocal rheo-scope, combination of confocal microscope and  
91 rheometer, has also been reported by Besseling et al. [8] which possessed rotational shearing

92 mode. However, the size, weight (greater than 10 kg) and cost of this device seem to have  
93 posed serious issues for its commercialization. The benefit of rotational motion over  
94 translation is the generation of an infinite shear and deformation. However, using rotational  
95 mode has the technical drawback of a compromised field of vision since the axis of motor  
96 rotation comes in line with the axis of observation through microscope objective. Use of  
97 translational motion overcomes this disadvantage because the axis of observation is at a right  
98 angle to the axis of motion during shearing action between *e.g.* parallel plates [10]. Wu et al.  
99 [17] developed a laboratory prototype of shearing cell based on translation mode. It was  
100 adapted to the inverted commercial CLSM device and possessed high accuracy from  
101 mechanical point of view.

102 Recently, a novel parallel plate rheological device (RheOptiCAD<sup>®</sup>) was designed by CAD  
103 Instruments and reported by Boitte et al. [10]. With this device a video recording during  
104 shearing is obtained from which the structural changes in the samples can be studied (changes  
105 in floc size, etc.). The device has primarily been designed to be mounted on an inverted  
106 microscope. Even though it can be used to visualize readily opaque structures [9], the device  
107 is not suited to analyse the optical behaviour of very concentrated clay suspensions (our topic  
108 of interest) as it makes use of transmitted light. This is why we proceeded to design an  
109 alternative set-up that would enable the study of these type of suspensions.

110 In the remainder of the article, we present the modified version of RheOptiCAD<sup>®</sup> with  
111 microscope system suited to analyse the behaviour of complex systems, particularly  
112 suspensions, under temperature-controlled conditions by shearing parallel-plate geometry.  
113 The design and specifications of the selected components are detailed. The set-up has been  
114 validated thanks to the pilot experiments. These experiments are done using un-flocculated  
115 and flocculated model kaolin suspensions. Additional experiments done on natural mud  
116 samples are also presented.

## 117 2. Design Considerations

118 The setup of our rheo-optical device makes use of the RheOptiCAD<sup>®</sup>, a parallel plate shearing  
119 device, commercially available from CAD Instruments (Illiers Combray, France) and  
120 introduced in 2012 [10] that can easily be coupled to a microscope. In short, the  
121 RheOptiCAD<sup>®</sup> device has been designed to enable optical analysis under a linear shearing  
122 force. During the measurement, a strain is applied in continuous, step or oscillation mode and  
123 a video recording is obtained. The analysis can be done in a temperature-controlled  
124 environment. The position of the shear plane in the cell can be adjusted by varying the  
125 velocity of the top and bottom plates.

126 Depending on the microscope used, an application-specific set-up involving the  
127 RheOptiCAD<sup>®</sup> and this microscope must be designed. In previous applications, the subject of  
128 study often was a heavily textured colloidal system like dough [10, 27], with high viscosity  
129 and being opaque. Therefore, the RheOptiCAD<sup>®</sup> device was used with an inverted confocal  
130 laser scanning microscope and fluorescent markers were used to visualize the changes in  
131 dough structure. Our aim is to study concentrated colloidal suspensions under shear  
132 accounting for the following constraints:

- 133 • The visualization of concentrated (very opaque) colloidal suspensions require the use  
134 of an upright microscope with an epi-illumination.
- 135 • Even in the presence of a sedimentation layer, the sample thickness should be much  
136 larger than the size of coagulated clay particles (0.1 mm). Therefore, microscope  
137 objectives with a large working distance (W. D.) should be selected. A simultaneous  
138 design constraint is that the objectives must have an (adjustable) cover slip correction.



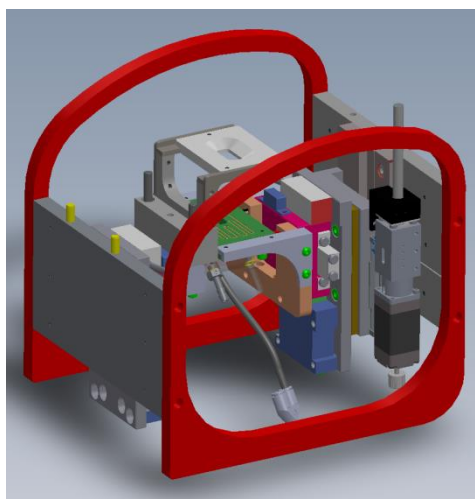
139 • To study clay particles with their "native" charge, addition of fluorescent marker  
140 molecules is unwanted. Therefore, the microscope should operate with direct  
141 illumination.

142 In the following sections we present a complete functional design and technical  
143 implementation, which enables this new application of the RheOptiCAD<sup>®</sup> system.

### 144 3. Functional Design

#### 145 3.1. Modification of the RheOptiCAD<sup>®</sup>

146 The RheOptiCAD<sup>®</sup> is manufactured by CAD Instruments (Illiers-Combray, France). The  
147 instrument is a modular design, contained in a cube-shaped frame with dimensions of about  
148 20 x 20 x 20 cm and total weight of about 5 kg. The construction is designed for optimal  
149 stiffness, which is achieved by using an aluminium construction with rounded corners and a  
150 minimal number of bolted joints (Fig. 1). The device can be (re)positioned in a microscopic  
151 setup by means of the "handles" that form the device's skeleton. This leaves the relative  
152 positioning of the internal parts unchanged. Within the cube-shaped frame, three motorized  
153 linear-stages are mounted. These stages can move under closed-loop control and are  
154 computer-controlled. Furthermore, the temperature of the device can be actively controlled by  
155 a Peltier cooler in the bottom plate, (green in Fig. 1) which is also under computer control.



156

157 FIG. 1. 3D image of the RheOptiCAD<sup>®</sup> rheometric device, modified for observation from above. The  
158 microscope objective is lowered into the recessed area on the top plate for observation of particles in the sample  
159 cell between top and bottom plate.

160 The design of the top and bottom plate of the RheOptiCAD<sup>®</sup> was adapted for observation  
161 from above. An opening with an oval shape was made in the top plate, to accommodate the  
162 microscope objectives (18 mm x 8 mm = 144 mm<sup>2</sup>), defining the dimensions of the  
163 observation window. Also, the vacuum ports in the top plate, necessary for the suction that  
164 keeps the glass slide in position, were moved to the side. In this way, sufficient horizontal and  
165 free vertical movement of the objectives was achieved. In the design presented here, with an  
166 upright microscope using epi-illumination and observation of reflected light, the displacement  
167 of the top plate is limited: the top plate can move with an amplitude of 12 mm, the bottom  
168 plate can travel as far as 20 mm.

### 169 **3.2. Microscopy subsystem**

170 For the upright microscope in the setup, we have found suitable components in the Olympus  
171 BXFM-BX3M modular microscopy/illuminator system [34]. From this modular system we  
172 have chosen the following parts:

- 173 • BX-FM-F Focusing unit, 30 mm range, 2 μm resolution
- 174 • Märzhäuser MFD motorised Z-stage drive mounted on the fine focusing knob[35]
- 175 • BX3M-KMA-S Epi-illuminator with white LED source
- 176 • U-5RE-2 5-fold nosepiece, equipped with:
  - 177 ○ LUCPLFLN 20x objective, coverslip correction (CC) 0-2 mm, W. D. 6.6-7.8
  - 178 mm, NA 0.45
  - 179 ○ LUCPLFLN 40x objective, coverslip correction (CC) 0-2 mm, W. D. 2.7-4.0
  - 180 mm, NA 0.6

- 181 • U-TLU-2 Telan lens-unit (tube lens)
- 182 • U-TV1X-2-7 1x video-adapter
- 183 • U-CMAD3-1-7 C-mount adapter ring

184 An impression of the microscopy subsystem is given in Fig. 2.

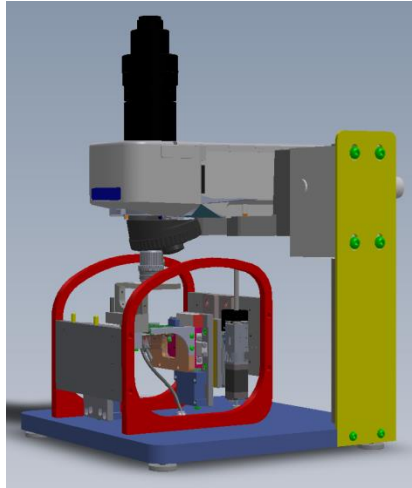


185

186 FIG. 2. The Olympus BXFM modular microscope with focusing unit (courtesy of Olympus). This microscope  
187 uses an LED white light source for broadband epi-illumination. Observation of the image in reflected light is  
188 done with a CMOS camera on top of the setup (not shown)

### 189 3.3. System assembly

190 The RheOptiCAD<sup>®</sup> skeleton and the microscope subsystem were assembled on a solid  
191 aluminium baseplate with adjustable feet for levelling. The focusing unit was attached to a  
192 solid stainless-steel rod with a diameter of 32 mm, according to Olympus factory  
193 specifications. The height was adjusted and fixed with an aluminium locking plate with six  
194 M6 Allen bolts (Fig. 3).



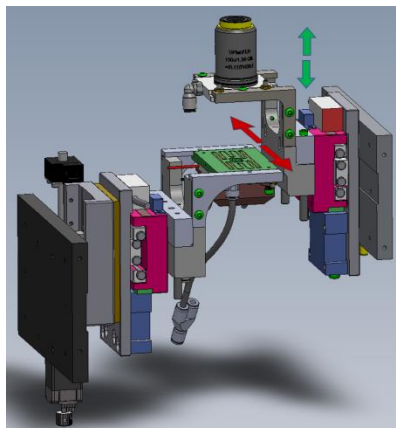
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FIG. 3. The assembled modified RheOptiCAD<sup>®</sup> setup.

196

### 197 3.4. Mechanical Control

198 In the RheOptiCAD<sup>®</sup> device, the shearing of the sample is carried out by uniaxial translation,  
199 generated by the motion of two parallel (top and bottom) plates. Each plate is driven by its  
200 own linear stage (Nanomotion FB-075 with HR4 piezo-electric motor) (NanoMotion,  
201 USA)[36]. The translational mode of shearing enables to make use of the rectangular shape of  
202 commercial microscopy glass slides, which makes the sampling user friendly. A vertical  
203 translation with a third motorized stage enables easy separation of the top and bottom plates  
204 for sample loading (Fig. 4). The sample is placed on a rectangular microscopy cover slip (24 x  
205 60 mm) attached to the bottom plate.



206

207 FIG. 4. Shear movement of the top and bottom plates of the RheOptiCAD<sup>®</sup> device (red arrows). Vertical  
208 movement of the bottom plate assembly enables loading of a suspension droplet on the bottom plate as well as  
209 adjustment of the cell height (green arrows)

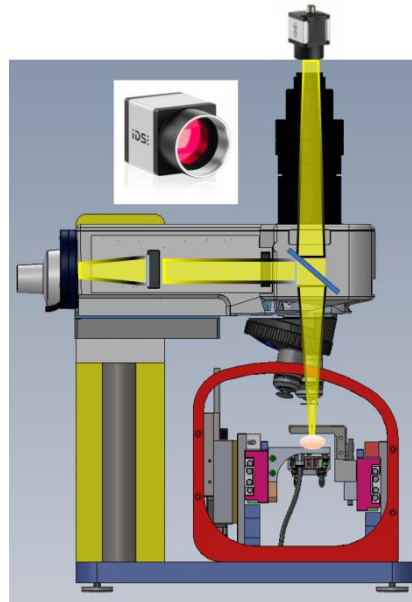
210 To ensure the parallelism and planarity of microscopy glass slides, important for better  
211 observation and fine-tune controlling of deformation, the glass slides were bound to the  
212 aluminium plates (top and bottom) by creating vacuum between them. A path for air was  
213 imprinted on the surface of both plates and the output point was connected to the vacuum  
214 pump (LaboPort KNF, France) having a minimum pressure limit of 160 mbar [10].

215 Before measurement, the bottom plate is raised until the sample is in contact with both plates.  
216 Subsequently, the distance between the plates (gap width) is adjusted to the desired value,  
217 thereby enclosing the droplet. All axes of translation are motorized with closed-loop control.  
218 The absolute encoders, Renishaw (RGH24Y15D30A), with 10 nm resolution used for the  
219 horizontal translations retain their position information. The vertical encoder is recalibrated in  
220 the RheOptiCAD<sup>®</sup> software when the top and bottom glass cover slips are replaced [10]. The  
221 motor control and recording of the plate's position are done by a modular motor control  
222 device, (Galil DMC 4040 [37]), which also generates a trigger pulse to start camera image  
223 acquisition.

224 Focusing of the objective is performed by the stepper motor in the Märzhäuser MFD  
225 motorised Z-stage drive [35]. Manual handling of the focusing knob is replaced by control  
226 from the computer user interface. At start-up, the focusing unit is driven to its top position,  
227 indicated by a micro switch. In this way, the focusing unit is calibrated.

### 228 **3.5. Optical image acquisition**

229 The optical layout of the microscope system is presented in Fig. 5. The LED source, at the  
230 back of the setup, provides white light. The source is imaged at the back focal plane of the  
231 objective (Köhler illumination). The light is focused on the sample by the objective.



232

233 FIG. 5. The optical layout of modified RheOptiCAD<sup>®</sup> device. A 1” CMOS camera (see insert) is fitted on the  
234 epi-illumination module of the microscope.

235 We have chosen plan-fluorite objectives for this assembly, for the following reasons:

- 236 • planarity of the field of view is essential
- 237 • colour correction as provided by plan-apochromatic lenses is not necessary for our  
238 experiments. We want to observe, locate and track particles under shear; therefore, we  
239 use a monochrome camera for optimal resolution.
- 240 • availability and affordability of long-working distance objectives with cover slip  
241 correction

242 The objectives mentioned above provide a working distance which is suitable for the 0-5 mm  
243 gap width of the RheOptiCAD<sup>®</sup>. Furthermore, the coverslip correction ring enables  
244 improvement of the image of particles near the bottom of the sample.

245 The objective collects the reflected light from a horizontal slice in the sample. With the  
 246 infinity-corrected optics chosen for this setup, a sharp image is created by a tube lens between  
 247 the semi-transparent mirror and the camera. The focal length of this tube lens determines the  
 248 image size on the camera. The Olympus tube lens and C-mount adapter for a 1" digital  
 249 camera was chosen.

250 The camera selected was a USB 3.0 connected monochrome CMOS camera with a 1" target  
 251 (12.5 x 10 mm) and a resolution of 2592 x 2048 pixels (UI-3180CP-M-GL Rev.2, IDS  
 252 GmbH, D). This camera is based on a PYTHON5000 CMOS-chip (ON Semiconductor) [38]  
 253 with some interesting features for microscopy:

- 254 • square 4.8  $\mu\text{m}$  x 4.8  $\mu\text{m}$  pixels for undistorted images
- 255 • a global shutter with various external trigger options via the control connector, this  
 256 enables synchronization with the motion of the RheOptiCAD<sup>®</sup> plates.
- 257 • choice of a Reduced Area-of Interest (ROI) and increased framerate. This opens the  
 258 option of using the camera at a resolution of 2048 x 2048 pixels, matching the circular  
 259 (flat) field of view of the microscope. Depending on the USB connection, framerates  
 260 of more than 80 Hz can be achieved.
- 261 • a CMOS sensor, apart from high resolution and high frame rate, provides reduced  
 262 blooming and smearing compared to CCD devices. This is an advantage in particle  
 263 tracking and analysis. Table 2 compares the features of the previous and current  
 264 versions of the RheOptiCAD.

265 Table 2: Differences between the two versions of the RheOptiCAD systems

Component name	RheOptiCAD <sup>®</sup> [8]	Our device
Modular upright microscope		X
Inverted microscope	X	

CMOS camera with square pixels and reduced blooming and smearing		X
CCD camera	X	
Epi-illumination		X
Vacuum joining of glass slides	X	X
Peltier system for temperature control	X	X

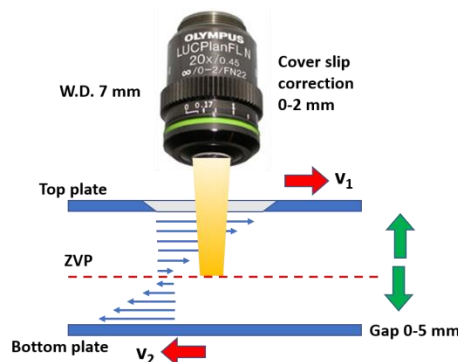
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### 267 3.6. Zero velocity plane

268 To analyse the structural changes in the sample under shear within the observation window, it  
 269 is essential for the object to be in the zero-velocity plane (ZVP). The position of this ZVP  
 270 between the plates in z-axis can be changed just by playing with the velocities of top and  
 271 bottom plates (see Fig. 6), according to the following equation:

$$272 \quad z_0 = \frac{e}{\frac{v_2}{v_1} + 1} \quad (3)$$

273 with  $z_0$  being the position of the ZVP in the z-axis direction, with the origin just underneath  
 274 the top plate.  $v_1$  and  $v_2$ , respectively, the velocities of the top and bottom plates ( $\text{mm s}^{-1}$ ), and  
 275  $e$  the gap width (mm).



276

277 FIG. 6. Movement of top and bottom plates along x-axis to change the location of ZVP (red arrows). Green arrows represent  
 278 the vertical movement (z-axis) of bottom plate for loading the sample and height adjustment



### 279 **3.7. Temperature control**

280 Temperature could be controlled within the range of 10-80°C using a Peltier system TEC-  
281 1090 Controller/Peltier Driver (Meerstetter Engineering, CH) (30 mm × 30 mm) mounted on  
282 the bottom plate. Water circulation, in the copper part of Peltier system, was used to regulate  
283 the sample temperature and a thermistor was fitted to the aluminium body of the bottom plate  
284 to monitor the temperature. Heating and cooling rates were optimized within the range of 1-  
285 20°C min<sup>-1</sup> using the PID controller.

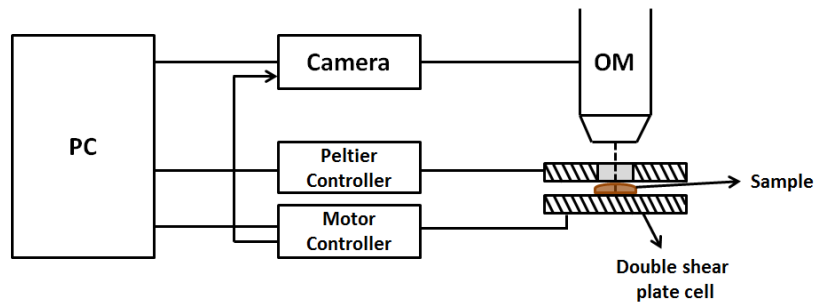
### 286 **3.8. Control software**

287 To make a user-friendly shearing device, a software was developed by CAD instruments,  
288 comprising of graphic interface, to define and control all the parameters related to the plates  
289 like, position of plates, mode of shear strain, velocity, amplitude, experiment time, frequency  
290 and gap width. Three modes of shear strain, after positioning the sample and setting the gap  
291 width, are available:

- 292 1. Step-strain for sudden deformation by having amplitude of each plate as variable (both  
293 plates are mobile)
- 294 2. continuous strain for linear deformation by having amplitude of each plate and  
295 experimental time as variables (both plates are mobile)
- 296 3. oscillatory strain for sinusoidal deformation by having amplitude and frequency of one  
297 of the plates as variable (one plate is mobile)

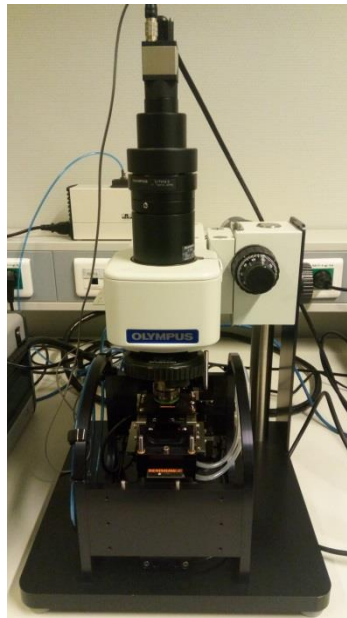
298 After each experiment, a data file is designed to be created automatically, which consists of  
299 the data supplied by the encoders of motorized stages. Different variables like position, time  
300 and velocity of top plate, bottom plate and gap width are recorded. Galil DMC-4040  
301 acquisition system allows the recording of data points. Fig. 7 shows the scheme of complete

302 software configuration and Fig. 8 shows the developed shear cell with an upright optical  
303 microscope.



304

305 FIG. 7. Schematic representation of complete software configuration; PC = personal computer; OM = optical  
306 microscope. The motor controller triggers continuous camera acquisition when the shear motion starts.



307

308 FIG. 8. Shear cell combined with an upright optical microscope and a camera

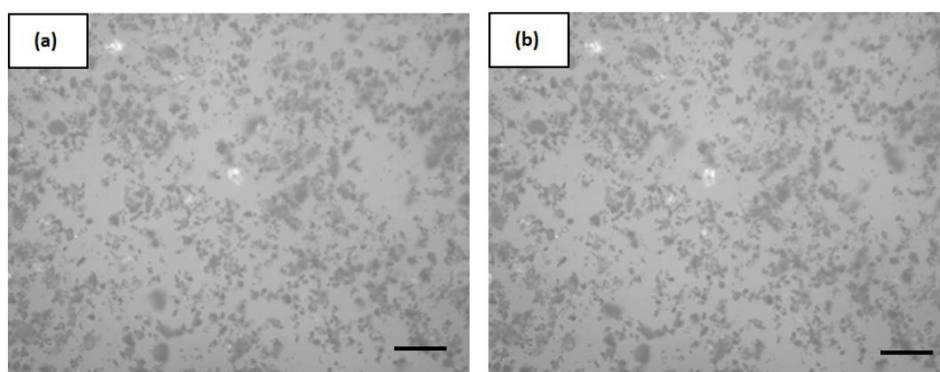
## 309 4. Validation Experiments

### 310 4.1. Kaolin suspension

311 The validation of the new set-up was performed with kaolin suspensions in water. Un-  
312 flocculated kaolin suspensions were prepared by dispersing small amount of kaolin (Imerys,  
313 England) in distilled water. Two commercial polyelectrolytes, Zetag 4120 (anionic copolymer  
314 of acrylamide and acrylic acid) and Zetag 8125 (cationic copolymer of acrylamide and  
315 quaternized ammonium cationic monomer) were used to prepare flocculated kaolin  
316 suspensions, by simply dispersing small amounts of polyelectrolytes and kaolin in distilled

317 water. For the optical analysis, we used our new optical microscope equipped with the 20x  
318 objective, having 0.45 NA and 6.6-7.8 mm of working distance. The gap between the two  
319 plates was varied from 100 to 10  $\mu\text{m}$  for different samples. In all cases, the sample was in  
320 contact with both upper and lower plates. This ensures that the samples were deformed  
321 instead of displaced. The temperature was maintained at 20°C for all the investigations. The  
322 LED light source was used and the images (2592 x 2048 pixels) were recorded in the x-y  
323 plane.

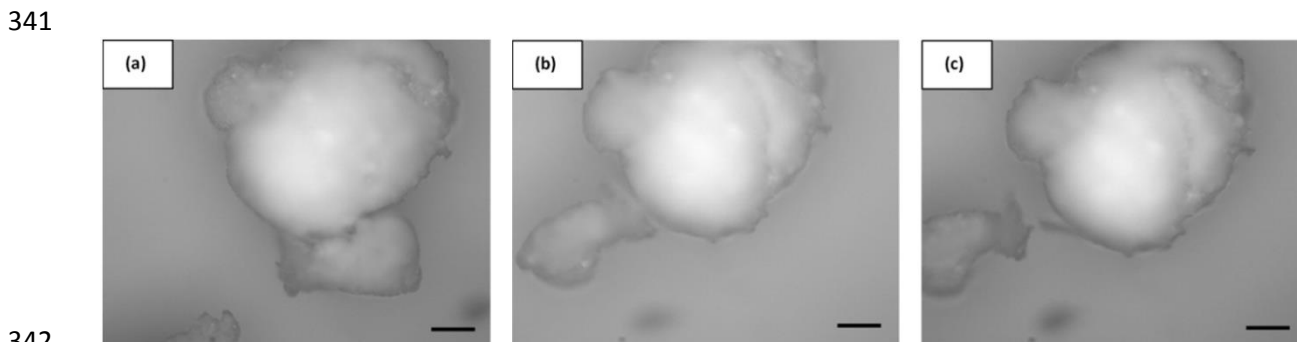
324 Several investigations were performed in oscillation mode using frequencies  $f$  between 0.5  
325 and 2 Hz and amplitudes  $A$  ranging from 0.1 to 0.5 mm for the bottom plate. The value of the  
326 amplitude was carefully chosen to make sure that the particles remain in the frame of view  
327 during the whole experiment. Fig. 9 shows the snapshots from the video recording for the un-  
328 flocculated kaolin suspension. It can be easily seen from the images that the unmodified  
329 kaolin particles are very small and homogeneously dispersed within the water. Under  
330 oscillatory shear, the particles showed a little bit movement due to the absence of any  
331 interactions between the particles.



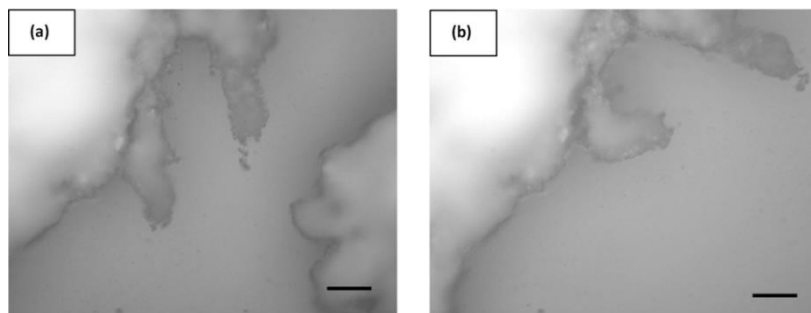
332  
333 FIG. 9. Images of un-flocculated kaolin suspensions subjected to oscillation at (a)  $t = 0$  s (b)  $t = 5$  s; Gap width = 10  $\mu\text{m}$ ;  $f =$   
334 0.5 Hz;  $A = 0.1$  mm. Slight movement of clay particles can be seen due to absence of interactions. Scale bar represents 70  $\mu\text{m}$

335 Figs. 10 and 11 present the images of the kaolin suspensions containing polyelectrolytes. All  
336 these images corroborate the formation of flocculated structures by addition of  
337 polyelectrolytes. Fig. 10 displays the break-up of a flocculated structure by the application of

338 a oscillatory shear for kaolin particles coated with cationic polyelectrolyte. Fig. 11b shows the  
339 stretching of a flocculated structure made of kaolin and anionic polyelectrolyte after 5 s of  
340 oscillatory shear at an amplitude of 0.5 mm and a frequency of 2 Hz.



342  
343 FIG. 10. Images of cationic polyelectrolyte-based kaolin suspensions subjected to oscillation at (a)  $t = 0$  s (b)  $t = 3$  s (c)  $t = 5$   
344 s; Gap width =  $100 \mu\text{m}$ ;  $f = 1$  Hz;  $A = 0.4$  mm. Sequence of images shows the break-up of flocs. Scale bar represents  $70 \mu\text{m}$

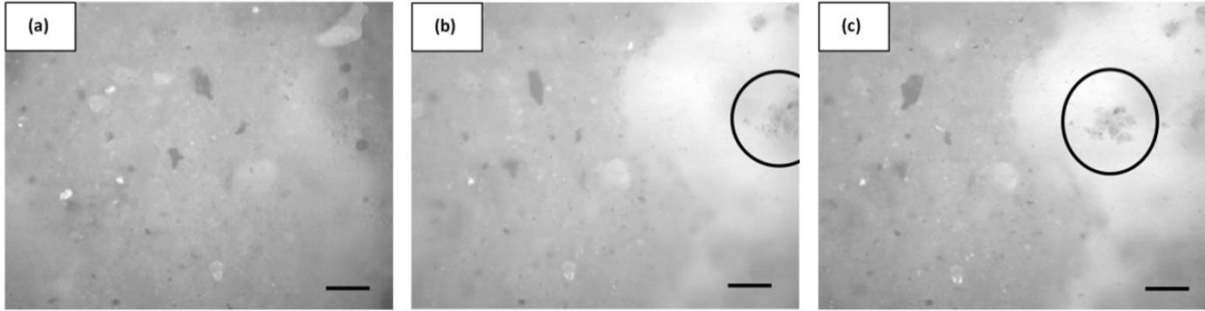


345  
346 FIG. 11. Images of anionic polyelectrolyte-based kaolin suspensions subjected to oscillation at (a)  $t = 0$  s (b)  $t = 5$  s; Gap  
347 width =  $100 \mu\text{m}$ ;  $f = 2$  Hz;  $A = 0.5$  mm. Second image presents the stretching of flocs after 5 s. Scale bar represents  $70 \mu\text{m}$

#### 348 4.2. Natural mud sample

349 A natural mud sample, collected from Port of Hamburg (Germany), was chosen for the  
350 investigation. The natural mud samples were placed on the bottom plate of the device and a  
351 gap of  $100 \mu\text{m}$  was set to perform the experiments at  $20^\circ\text{C}$ . Firstly, the samples were  
352 subjected to continuous strain by setting the movement of both plates in opposite directions.  
353 The images selected from the video recording (which can be found in the online  
354 supplementary material) are shown in Fig. 12. Fig. 12b shows the breakage/separation of a  
355 bigger flocculated structure into two flocs which further divided into more smaller flocs as  
356 shown in Fig. 12c.

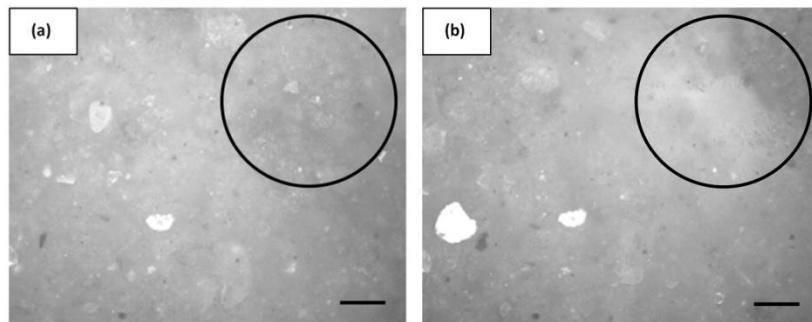
357



358

359 FIG. 12. Images of a natural mud suspension subjected to continuous strain at (a)  $t = 0$  s, (b)  $t = 5$  s, (c)  $t = 10$  s. Sequence of  
360 images shows the structural break-up during shear. The black circle shows a small floc that has detached from a bigger one  
361 on the right (not in view). Scale bar represents  $70 \mu\text{m}$

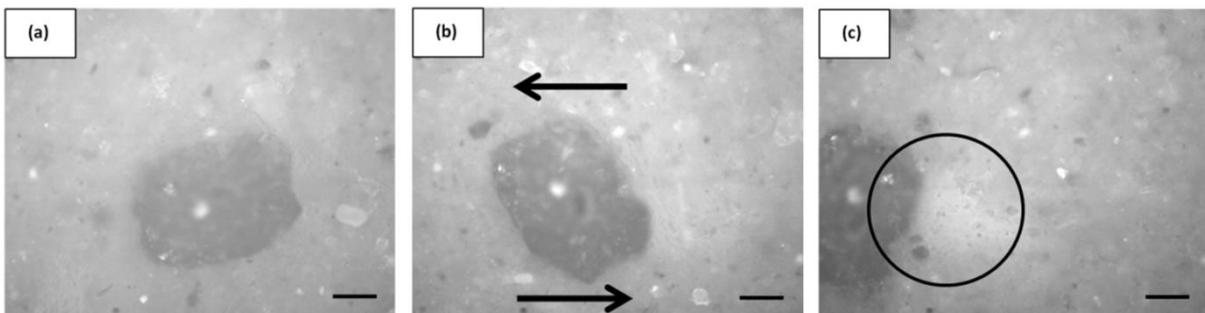
362 The optical shearing of the samples was also performed in oscillation mode by oscillating the  
363 bottom plate at 1 Hz with the amplitude of 0.4 mm. The images selected from the video  
364 recording are shown in Figs. 13 and 14. Fig. 13b shows the breakage of a flocculated  
365 structure after 3 s of oscillation motion. Fig. 14a shows the presence of a large particle (clay)  
366 in the suspension, which displayed the rotational motion during oscillatory shearing as  
367 indicated by the arrows in Fig. 14b. This large particle also creates a void during oscillation  
368 after 10 s, as shown in Fig. 14c.



369

370 FIG.13. Images of a natural mud suspension subjected to oscillation at (a)  $t = 0$  s, (b)  $t = 3$  s. The black circle shows the result  
371 of the breakage of bigger flocs by the presence of a void (white colour) in the second image. Scale bar represents  $70 \mu\text{m}$

372



373

374 FIG. 14. Images of a natural mud suspension subjected to oscillation at (a)  $t = 0$  s, (b)  $t = 5$  s, (c)  $t = 10$  s. Arrows shows the  
375 direction of rotational motion of particle during shearing. The white area inside the black circle represents a void created by  
376 the tumbling motion of the large particle. Scale bar represents  $70 \mu\text{m}$

## 377 **5. Conclusion**

378 This study presents the modification of an already reported rheo-optical device[10]. The  
379 modified system enables the observation of sedimentating and/or concentrated suspensions,  
380 by using an upright optical microscope configuration instead of an inverted one as in the  
381 original device. An optical microscope with epi-illumination using reflected light was used to  
382 get the optical signature of suspensions. Proof-of-concept experiments performed by using  
383 un-flocculated and flocculated kaolin suspensions and a natural mud suspension, confirmed  
384 the applicability of our device for investigating complex systems. Successive snapshots taken  
385 from the video recording of these suspensions under shear revealed the structural changes of  
386 these systems as a function of the shearing action. The new rheo-optical device will be used,  
387 in the future, to perform state of the art research in the field of sediment rheology by linking  
388 the qualitative structural break-up and build-up (thixotropy) of mud suspensions observed by  
389 the present device to the quantitative rheological measurements obtained from a conventional  
390 rheometer.

391 In future applications/research, some technical modifications can be done to optimize the  
392 presented device such as: [i] incorporating a device for stress measurement (material's  
393 response to the applied deformation), [ii] having a translation motion of plates in y-axis, in  
394 addition to x-axis, [iii] adding the possibility to have oscillation of both plates simultaneously,  
395 instead of one plate, in oscillation mode, [iv] modifying the surface properties of the glass  
396 slides in case of sticky materials which would also enable to study particle-wall interactions,  
397 and [5] improving the data analysis software for quantitative investigation.

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