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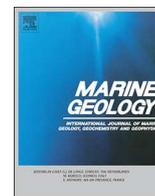
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Yield stress measurements of mud sediments using different rheological methods and geometries: An evidence of two-step yielding



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ABSTRACT

Yield stress materials have a wide range of commercial applications. Yet, the suitable way of determining the yield stress values of a given material has been the subject of many studies and debates. Yield stresses are dependent on the material (shear) history and composition, which implies that robust protocols should be developed to study the yield stress dependence on a given parameter. In this study, three natural mud samples from a port having different densities were chosen for analysis. Four different geometries including concentric cylinders (Couette), cone and plate, parallel plates, and vane geometries were used. Our aim was to find the geometry and measurement protocol that best adapted to natural mud samples: the measurement should be reasonably fast and the major changes in sample structure (two-step yielding) should be recorded within the same measurement. Various rheological experiments such as stress sweep, oscillatory amplitude sweep, creep and stress growth tests were tested. Two-step yielding behavior was observed for the mud samples in stress sweep and amplitude sweep tests. The first yield point was linked with the breakage of interconnected network of aggregates/flocs while the second one was attributed to the collapse of aggregates into the smaller flocs or individual particles. Stress sweep tests proved to be practical, time efficient, and reliable tests for measuring yield stress values. Our study showed that Couette and parallel plate geometries are the most suitable geometries for analyzing the two yield stresses of the samples. Vane geometry is appropriate to study consolidated (solid-like) systems as for these samples a Couette geometry cannot be used because the bob could get stuck during the experiment.

1. Introduction

Composite materials can exhibit either solid-like or liquid-like properties when submitted to an applied stress. These materials then behave like an elastic solid at small stresses whereas they tend to flow above a critical value of stress, called yield stress. There are lots of examples of such systems including cosmetic creams, toothpaste, margarine, polymeric gels, and colloidal suspensions (Bird, Dai, and Yarusso, 1983; Clayton, Grice, and Boger, 2003; Yoshimura and Prud'homme, 1988). Yield stress determination is very important for industrial processes. Yield stress measurements are used for instance to determine the minimum pressure in a slurry flow in a pipeline, to analyze its stiffness or to examine the possibility of air entrapment within its structure. Our overall goals are to study the yield stresses of mud sediments and to propose an appropriate geometry and rheological

method for these systems. Yield stress has been found to be an important parameter to define navigable fluid mud layers for ports and waterways (Kirichek et al., 2018; Møller, Mewis, and Bonn, 2006; Nguyen and Boger, 1992). Our present study is imbedded in a larger scale project which aims at correctly assessing the so-called nautical bottom within navigable channels.

The term yield stress was first introduced by Bingham and coworkers for plastic yielding in metals (Barnes, 1999; Bingham, 1922). From the last two decades, a huge interest in yield stress materials has led to the development of several experimental techniques for estimating yield stress values. However, these values can vary more than one order of magnitude depending upon the selected method and the handling procedure of sample before measurements (James, Williams, and Williams, 1987; Nguyen, Akroyd, De Kee, and Zhu, 2006; Steffe, 1996; Uhlherr et al., 2005; Zhu, Sun, Papadopoulos, and De Kee, 2001).

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This variability in yield stress values is usually attributed to the differences in principles associated with the experimental methods, criterion of yield stress definition and the time scale of the chosen experiment (Cheng, 1986). Apart from different rheological methods, several geometries including concentric cylinder (Couette), parallel plate, cone & plate and vane type, are also available to perform rheological measurements. Each geometry has its own merits and limitations, which makes it suitable for wide range of complex systems. The vane geometry has become quite popular for yield stress measurements due to its simplicity and slip prevention during shearing action (Assaad, 2018; Saak, Jennings, and Shah, 2001), particularly for determining the shear strength of soils (ASTM, 2008).

Recently, it has been stated that, instead of a single yielding response, a prominent two-step yielding behavior was observed for different soft materials. This two-step yielding suggested the existence of structural level of different length scales, due to the different types of interactions. Experimental evidence for this interesting two-step yielding phenomenon has been found in literature for various systems including colloidal glasses (Pham et al., 2008), carbopol microgel (Shao, Negi, and Osuji, 2013), mud sediments (Shakeel et al., 2020), colloidal gel (Chan and Mohraz, 2012), magneto-rheological systems (Segovia-Gutiérrez, Berli, and Vicente, 2012), muscovite dispersions (Nosrati, Addai-Mensah, and Skinner, 2011), etc. using different rheological tests. The most frequently used experiment, to investigate the two-step yielding, is to perform the oscillatory amplitude sweep test by increasing either stress or strain amplitude and recording the material's response in terms of storage (G') and loss (G'') moduli. The two-step yielding is typically recognized by the two distinct peaks in G' or G'' response, which are usually linked with the bond and cage breaking

process. It has also been reported in the literature that this two-step yielding behavior strongly depends on the solids volume fraction, ϕ . The experiments have revealed that the second yield point becomes less significant as ϕ decreases, until the system displayed only a single-step yielding behavior at $\phi \sim 0.2$ (Koumakis and Petekidis, 2011; Krumb and Zukoski, 2010). A brief overview of the different systems showing two-step yielding, along with their possible explanation of two-step yielding response is presented in Table 1.

An extensive rheological/ yield stress analysis of natural mud samples from different parts of the world using different geometries has been reported in the literature. Carneiro, Fonseca, Vinzon, and Gallo (2017) reported the rheological analysis of fluid mud samples from Port of Santos (Brazil) using vane geometry. The same geometry was also used to investigate the steady rheology of mud samples from Hangzhou Bay, Yangtze River and Yangcheng Lake in China, (Yang, Yu, Tan, and Wang, 2014a) and the Port of Santos, the Port of Itajaí, the Port of Rio Grande and the Amazon south navigation channel in Brazil (Fonseca, Marroig, Carneiro, Gallo, and Vinzón, 2019). Smooth parallel plate geometry was also reported in the literature to study the rheological response of natural mud samples from Yueqing Bay in China (Yang and Yu, 2018) and Hendijan Coast in the Persian Gulf (Soltanpour and Samsami, 2011) under steady and oscillatory shearing conditions. Huang and Aode (2009) examined the rheological fingerprint of the natural mud samples from two different locations of Hangzhou Bay (China) using cone and plate, and Couette geometries. Couette geometry was also used to observe the rheological properties of Chorfa dam mud in Algeria (Messouadi, Bouzit, and Boualla, 2018) and mud samples from Lianyungang in China (Xu and Huhe, 2016). Van Kessel and Blom (1998) presented the comparison of rheological properties of

Table 1

A brief overview of the literature on the two-step yielding systems.

| Investigated system | Experiments | Possible explanation for two-step yielding | Ref. |
|---|---|---|--|
| PMMA particles stabilized with PS dispersed in <i>cis</i> -decalin | Oscillatory amplitude sweep, stress growth, creep, flow curve | The first yielding was associated with the breaking of attractive bonds between the particles and the second yield point was linked with the cage breaking phenomenon. | (Pham et al., 2008) |
| Carbopol microgel | Oscillatory amplitude sweep, oscillatory time sweep | The first yielding was attributed to the network rupture followed by the cluster formation due to strain densification. The second yield point was associated with the breakage of these clusters. | (Shao, Negi, and Osuji, 2013) |
| Muscovite particles dispersed in water | Stress sweep | The first yield point was suggested to be linked with the breakdown of 3D network of particle aggregates. The second yielding was attributed to the rupture of aggregates into individual particles. | (Nosrati, Addai-Mensah, and Skinner, 2011) |
| CoNi nanoplatelet dispersed in castor oil | Oscillatory amplitude sweep | The first yield point was associated with the inter-cluster bond breaking followed by the densification, while the second one was linked with the cluster breaking process. | (Arief and Mukhopadhyay, 2019) |
| Anionic surfactants, clay, and abrasive particles of calcite dispersed in water | Oscillatory amplitude sweep | The first yielding was attributed to the network rupture, while the second one to the collapse of aggregates. | (Shukla, Arnipally, Dagaonkar, and Joshi, 2015) |
| Cocoa powder dispersed in the mixture of vegetable oil and water | Oscillatory amplitude sweep | The first yielding point was associated with the fragmentation of network structure into large agglomerates, while the second yield point was attributed to the breakage of agglomerates into small fragments. | (Ahuja and Gamonpilas, 2017) |
| PMMA particles stabilized by PHSAs chains dispersed in <i>cis</i> -decalin | Oscillatory amplitude sweep, stress growth | In case of glasses, the first yield point was attributed to the bond breakage and the second one to the cage breaking. In case of gels: the first yield point was attributed to the bond breakage while the second one to the cluster breaking. | (Koumakis and Petekidis, 2011) |
| PMMA particles stabilized by PHSAs chains dispersed in the mixture of <i>cis</i> -decalin and cycloheptyl bromide | Stress growth | Two-step yielding was linked with the existence of cages of two different sizes (i.e., two different length scales). | (Sentjabrskaja et al., 2013) |
| Mixture of PS core/PNIPAM shell microgels and PSS particles dispersed in water | Oscillatory amplitude sweep | The first yield point was attributed to the breakage of interconnected clusters and the second one was linked with the further collapse of clusters. The incorporation of PSS particles weakens the network structure and converges the two-step yielding into a single step. | (Jia, Hollingsworth, Zhou, Cheng, and Han, 2015) |
| Mud sediments from port of Hamburg, Germany | Stress sweep, oscillatory amplitude sweep, stress growth, creep | The first yield point was linked with the breakage of interconnected network of flocs while the second one was attributed to the collapse of aggregates/flocs into the smaller flocs or individual particles. | This study |

PMMA = poly methyl methacrylate; PHSAs = polyhydrostearic acid; PS = polystyrene; PNIPAM = poly(*N*-isopropylacrylamide); PSS = sulphonated polystyrene.

natural and artificial mud samples using three different geometries: (i) Couette, (ii) double concentric and (iii) cone and plate. They found in particular that their results were dependent on the geometries used in the tests. Despite the numerous studies on the rheological properties of natural mud samples, a systematic comparison between all available geometries as function of the properties (composition) of the samples is still missing. In the present study, we aim at finding the geometry that will give us all the major changes occurring in sample structure upon shearing.

Concerning the rheological protocols, [Claeys et al. \(2015\)](#) proposed a protocol to be used with a vane-type of rheometer to investigate the rheological behavior (i.e., equilibrium flow curve) of mud samples. Their main aim was to develop a protocol to obtain equilibrium flow curves with good repeatability, from which the dynamic yield stress of the disturbed sample can be deduced. Conventional methods to measure yield stresses are steady stress sweep, oscillatory amplitude sweep, creep and stress growth experiments ([Coussot, 2014](#); [Dinkgreve, Paredes, Denn, and Bonn, 2016](#); [Nguyen and Boger, 1992](#)). However, a detailed comparison between these methods using different geometries, to effectively analyze the two-step yielding behavior of mud sediments is not available in the literature. Therefore, we aim at (i) finding the suitable rheological method and geometry that will ensure a fast and reliable measurement of the changes in sample structure, and their corresponding yield stresses and (ii) investigating the two-step yielding behavior of mud sediments, having different densities, using different rheological tests. For these objectives, natural mud samples of different densities were collected from Port of Hamburg.

2. Experimental

2.1. Materials

In this study, natural mud samples were taken from the Köhlfleethafen area in the Port of Hamburg (Germany) using 1 m cores. The collected samples were divided into three different layers based on the differences in their visual consistency: fluid mud (FM), pre-consolidated sediment (PS) and consolidated sediment (CS) ([Fig. 1a](#)). The location was chosen on the basis of a preliminary analysis which showed that the samples from the selected location were quite stable and have an intermediate amount of organic matter compared to other locations. The samples were packed in a sealed container and shipped to the laboratory. The dry density of the minerals was taken to be 2650 kg/m^3 . The bulk density of the sediments was determined by the method reported elsewhere ([Coussot, 2017](#)). In short, the mass of the mud samples was determined, before and after oven drying at $105 \text{ }^\circ\text{C}$ for 24 h. This gave the mass of the dry solids and water content in the

mud samples. By using the densities of water and minerals, the corresponding volumes were then estimated. The final bulk density of mud samples was then calculated based on these masses and volumes.

Particle size distribution of the mud samples was measured using static light scattering technique (Malvern Mastersizer 2000MU) by extensively diluting the mud samples. [Fig. 1b](#) displays the particle size distribution of the three selected mud layers. The organic matter content of the samples was determined using an ISO standard 10,694:1996–08 ([ISO., 1995](#)). The characteristics (particle size, bulk density and organic matter content) of all the mud layers are summarized in [Table 2](#). Before the rheological experiments, all the mud samples were gently homogenized by hand stirring.

2.2. Equipment

The Thermo Scientific HAAKE MARS I rheometer was used to perform the rheological measurements. This rheometer can either be operated in stress controlled, shear rate controlled or deformation controlled mode. Controlled shear stress (CSS) mode of the rheometer was used to perform stress sweep tests, oscillation amplitude tests, and creep tests while controlled shear rate (CSR) mode was selected to perform stress growth experiments. Four different geometries, including smooth and grooved concentric cylinders (Couette & Couette-G), cone and plate (CP), parallel plates (PP), and vane were used to perform different rheological tests. For concentric cylinder geometry, the cup inner and bob outer diameters were 27 mm and 25 mm, respectively. The distance between the bob and the bottom of the cup was 5.30 mm. In case of vane geometry, the cup inner diameter was 27 mm. Vane geometry having diameter of 22 mm was used by maintaining a distance of 1 mm between the vane and the bottom of the cup. Cone and plate geometry, having diameter of 60 mm with 2° cone angle and 0.104 mm gap between the cone and plate, was used to perform experiments. The diameter of parallel plates was 35 mm. A 2 mm gap was adjusted between the plates for rheological experiments. All the experiments were duplicated to evaluate the repeatability of the methods.

3. Methods

3.1. Stress growth

In a stress growth method, a constant shear rate is applied and the resultant shear stress is measured as a function of time. Typical stress growth curves initially display a linear regime followed by either a deviation from linearity or a stress overshoot and then finally a steady state value of stress ([Barnes and Nguyen, 2001](#)). Different yield stress values can be obtained from this method depending upon the definition

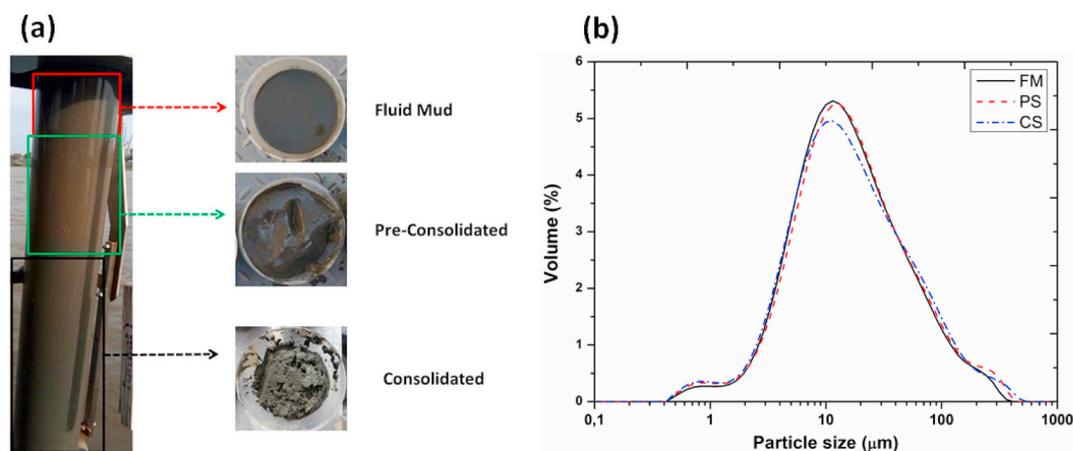


Fig. 1. (a) Different mud layers at the Port of Hamburg (Germany) (b) Particle size distribution of fluid mud (FM), pre-consolidated sediment (PS) and consolidated sediment (CS).

Table 2
Characteristics of different mud layers from Port of Hamburg (Germany).

| Mud layer | Bulk Density (kg/m ³) | Median particle size (D ₅₀) (μm) | Polydispersity/Span (–) | Organic matter (% TS ^a) |
|------------------|--------------------------------------|--|----------------------------|--|
| Fluid mud | 1134 ± 17 | 13.5 ± 0.2 | 4.5 | 3.7 |
| Pre-consolidated | 1158 ± 19 | 13.6 ± 0.3 | 4.7 | 3.4 |
| Consolidated | 1186 ± 18 | 14.2 ± 0.2 | 5.1 | 3.7 |

^a TS = Total solids

of yield point such as departure from linearity, peak stress, or steady state (equilibrium) stress value (Møller, Mewis, and Bonn, 2006). In our experiments, different shear rates ranging from 0.01 to 10 s⁻¹ were applied and the stress for 300 s was recorded, which was enough to capture the peak stress. The selected range of shear rate was sufficient to analyze the effect of the applied shear rate on the stress growth behavior.

3.2. Stress sweep

A stress sweep test is performed by linearly increasing the shear stress from zero to a value much higher than the yield stress, while the resultant strain is recorded. The result can be plotted either in the form of apparent viscosity as function of shear stress or strain vs shear stress. From the viscosity curve, the yield stress can be determined from the sharp decline in viscosity above the yield stress or by the sudden increase (non-linearity) in the slope of the strain curve. If the decline in viscosity or non-linearity in strain is not very sharp and clear, the point of intersection between the straight lines extending from the plateau where viscosity is more or less constant (structured state) and unstructured sample where everything is broken down (liquid-like state), can be used to determine the yield stress values (Zhu et al., 2001). Stress sweep tests for all tested geometries were performed using the controlled stress mode of the rheometer. An increasing stress ramp of 1 Pa/s was applied from 0 to 300 Pa. The upper/higher limit of the stress was chosen depending upon the consistency of the sample. The corresponding angular displacement was measured, and the shear rate and viscosity were then determined.

3.3. Oscillatory amplitude sweep

Oscillatory amplitude sweep experiments typically involve the application of a sinusoidal strain or stress at a particular frequency. The outcome of this method can be plotted in the form of storage modulus (G') and loss modulus (G'') curves as a function of either stress or strain. The storage (G') and loss (G'') moduli, obtained from oscillatory experiments, are the in-phase and out-of-phase responses of the material, respectively, to the applied sinusoidal stress/strain. Several ways of determining yield stress from oscillatory experiments have been suggested in the literature such as: (1) cross-over point between G' and G'' (Kugge, Vanderhoek, and Bousfield, 2011; Perge, Taberlet, Gibaud, and Manneville, 2014; Renou, Stellbrink, and Petekidis, 2010), (2) decline in storage modulus as a function of stress/strain, given by the point of intersection between the horizontal line representing the linear viscoelastic behavior and a line representing the non-linear behavior well above the yield point (De Graef, Depypere, Minnaert, and Dewettinck, 2011; Rouyer, Cohen-Addad, and Höhler, 2005), (3) by plotting the elastic stress as a function of stress/strain, where the elastic stress τ_E is defined by:

$$\tau_E = G'\gamma \quad (1)$$

At very small stress/strain a linear behavior is typically observed followed by a sharp decline in τ_E above yield point. The accuracy of these methods may depend on the applied frequency. Preliminary

amplitude sweep tests at different frequencies were performed in our case to analyze the suitable value of the frequency for amplitude sweep tests. In our tests, the stress amplitude was increased from 0 to 200 Pa depending on the density of the samples at a constant frequency of 1 Hz. The material's response was obtained in terms of G' and G'' as a function of the applied amplitude.

3.4. Creep

The creep test is another way to measure yield stress values (Christopoulou, Petekidis, Erwin, Cloitre, and Vlassopoulos, 2009). In a typical creep test, constant stresses in a range encompassing the yield stress are imposed in successive experiments to the sample and the resultant strain is measured as a function of time. Below the yield stress, the sample behaves like a solid with small strain which reaches a constant value with time. Above the yield stress, the strain shows a sudden increase and becomes an increasing function of time. The measurement time is important in this method to analyze the correct value of yield stress. Contrary to the other methods mentioned before, the creep method requires a prior knowledge of the range of yield stress, in which structural break-up (yielding) is expected. In our creep experiments, the applied stress values were chosen to be: (i) below first yield point, (ii) above first yield point, (iii) below second yield point and (iv) above second yield point, as determined by the stress sweep tests.

4. Results and discussion

4.1. Stress sweep tests

The rheological analysis of concentrated suspensions using rheological geometries such as Couette and parallel plate may encounter the problem of wall slip, particularly at low shear rate/shear stress (Barnes, 1995). The wall slip effect usually presents itself as difference in viscosity values for geometries with different sizes, two-step yielding instead of a single step, etc. The existence of wall slip can be easily recognized, for example, for the parallel plate geometry by using different gaps and comparing the results (Yoshimura and Prud'homme, 1988). This wall slip effect can also be identified by comparing the results of smooth and roughened/serrated geometries. In this study, both approaches have been used to identify the wall slip effect. In case of parallel plate, stress sweep experiments were performed with variable gaps and the results are shown in Fig. 2a. While for Couette geometry, grooved geometry was used to compare the obtained results of stress sweep with the smooth Couette, as shown in Fig. 2b. Both these approaches showed the absence of wall slip, as the results were more or less similar, for the fluid mud sample. The similar results, i.e., absence of wall slip, were also observed for other two samples (pre-consolidated and consolidated samples) (data not shown).

Stress sweep tests were used to determine the yield stress of our mud samples because of their wide applicability for yield stress measurements. Although the stress sweep method can provide reproducible results, yet we found that this method depends on the type of geometry used for the measurements. The apparent viscosity of the fluid mud

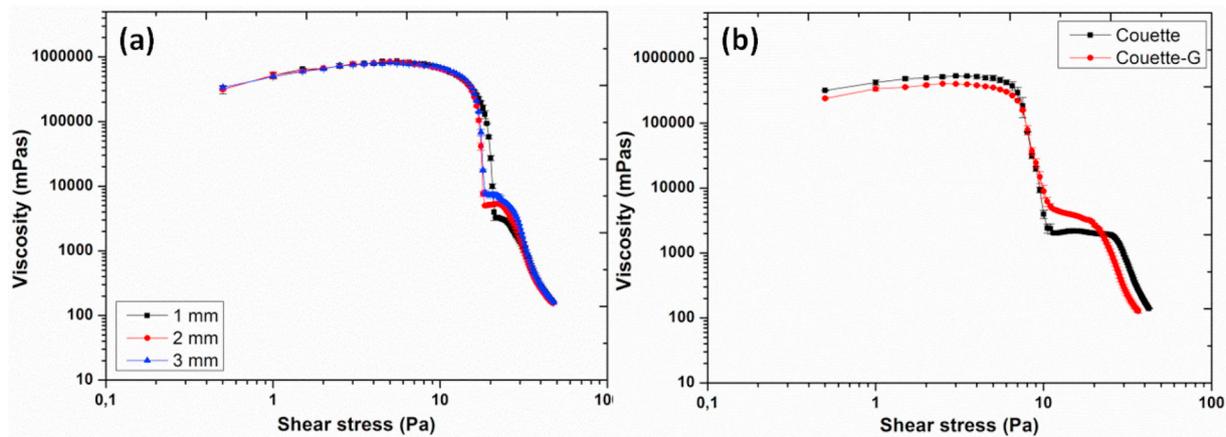


Fig. 2. Apparent viscosity as a function of shear stress for (a) parallel plate geometry with variable gap and (b) smooth and grooved Couette geometries for fluid mud layer. Bars represent standard deviation.

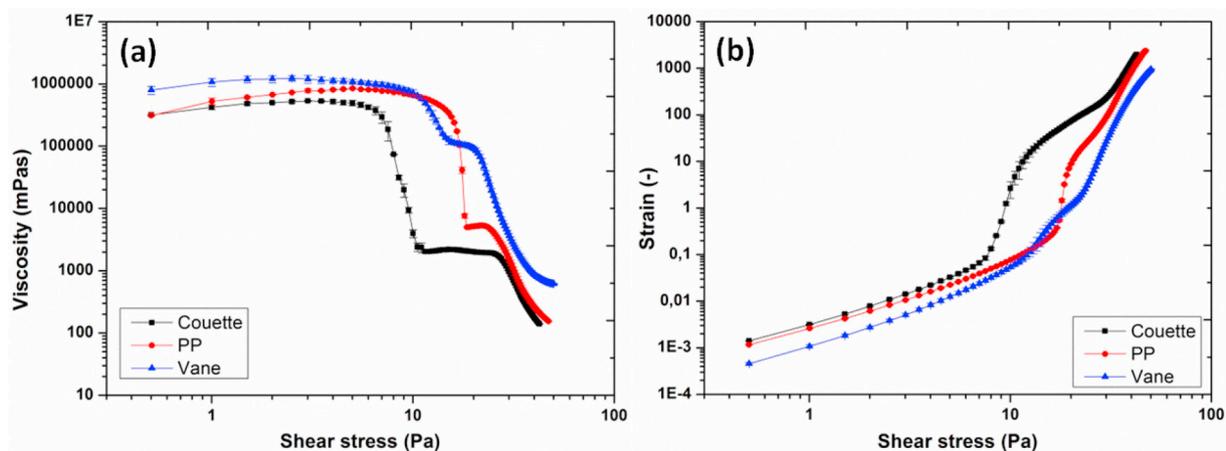


Fig. 3. (a) Apparent viscosity and (b) strain as a function of shear stress for fluid mud layer using different geometries; The solid lines are just a guide for the eye. Bars represent the standard deviation.

(FM) layer as a function of shear stress for different geometries is shown in Fig. 3a. From the viscosity curve, two-step yielding was identified from the decline in viscosity, for all three geometries. Cone and plate geometry was discarded for further experimentation because the response of the material was very scattered due to the presence of large particles within the small gap of cone and plate (data not shown). Pre-consolidated (PS) and consolidated (CS) mud layers displayed similar trends of apparent viscosities as a function of shear stress for different geometries (shown in Fig. S1a & S1c). In order to compare the yield stress values of different samples, the approach reported by Zhu et al., 2001 was used to obtain the yield stress values by extrapolation. The stress values associated with these two yield points are referred to as static yield stress, τ_y^s (first decline) and fluidic yield stress, τ_y^f (second decline).

The similar two-step yielding was also reported in literature for different systems such as colloidal glasses (Pham et al., 2008), carbopol microgel (Shao, Negi, and Osuji, 2013), colloidal gel (Chan and Mohraz, 2012), magneto-rheological systems (Segovia-Gutiérrez, Berli, and Vicente, 2012), muscovite dispersions (Nosrati, Addai-Mensah, and Skinner, 2011), etc. For example, Potanin (2019) reported the effect of two different polymers (xanthan gum and carboxymethyl cellulose (CMC)) on the two-step yielding behavior of silica dispersions. The results showed the convergence of two-step yielding into a single step by adding xanthan gum into silica dispersions while the two-step yielding phenomenon retained by the incorporation of CMC into the dispersion. The disappearance of two-step yielding in case of xanthan

gum was attributed to the integration of silica particles into the uniform network of xanthan gum.

The system investigated in the current study, i.e., mud sediments, also primarily composed of clay particles and organic matter/biopolymer. On the basis of existence of two-step yielding in mud samples, it can be stated that the organic matter/biopolymer in mud samples acted in the same way as CMC in silica dispersions, i.e., as a thickening agent without forming polymeric network. Furthermore, the first (static) yield stress value in mud sediments is suggested to be linked with the breakdown of interconnected network of flocs/aggregates. Whereas the further collapse of flocs/aggregates into smaller flocs or individual particles is associated to the second (fluidic) yield stress value, as similar explanation was also reported for silica dispersions (Potanin, 2019). However, the two-step yielding in CS samples may occur due to the formation of micro-cracks which results in thin water layer formation at the wall. It is also reported in literature that the cohesiveness and rheological properties of muddy sediments are strongly affected by the presence of small amounts of organic matter (Malarkey et al., 2015; Parsons et al., 2016; Paterson, Crawford, and Little, 1990; Paterson and Hagerthey, 2001; Schindler et al., 2015; Shakeel, Kirichek, and Chassagne, 2019). Apart from rheology, floc size and settling rates for muddy sediments or for sand/mud mixtures have also been analysed in the literature (Manning, Baugh, Spearman, Pidduck, and Whitehouse, 2011; Manning, Baugh, Spearman, and Whitehouse, 2010; Mehta, Manning, and Khare, 2014; Soulsby, Manning, Spearman, and Whitehouse, 2013; Spearman, Manning, and Whitehouse, 2011;

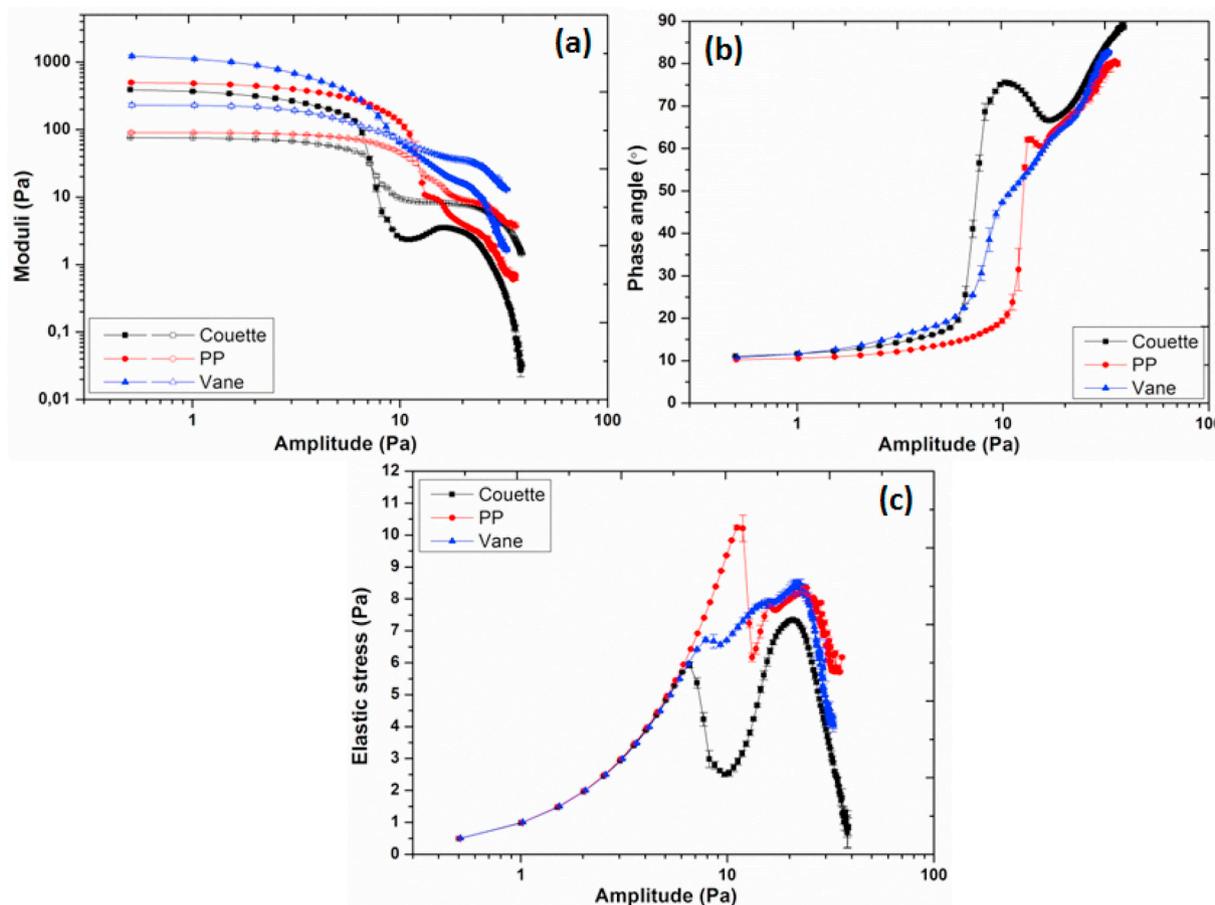


Fig. 4. (a) Storage modulus, (b) phase angle and (c) elastic stress as a function of amplitude for fluid mud layer using different geometries at 1 Hz. Solid line is just a guide for the eye. Bars represent standard deviation.

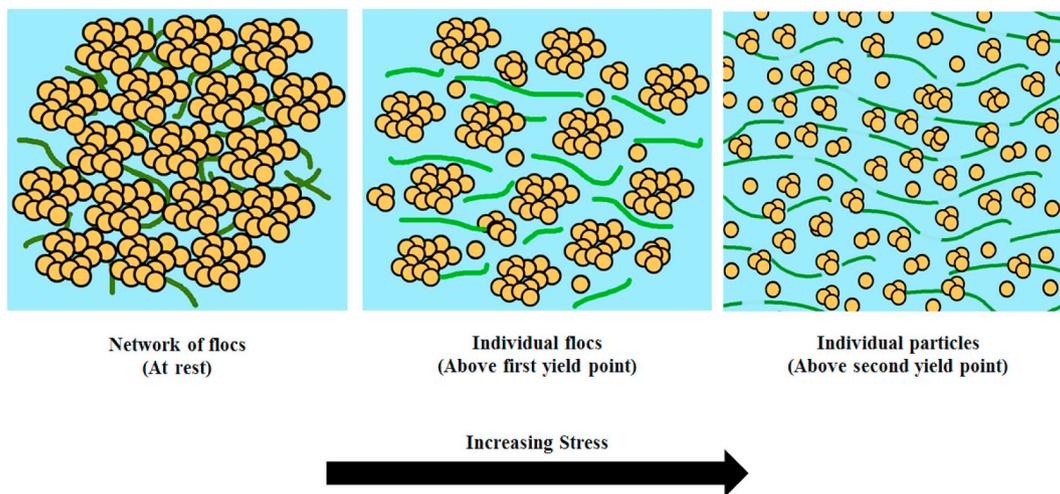


Fig. 5. Possible mechanism, proposed in literature (see Table 1), for two-step yielding in mud sediments. Yellow circles represent clay particles and green lines represent the organic matter. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Spencer, Manning, Droppo, Leppard, and Benson, 2010; Whitehouse and Manning, 2007; Whitehouse, Soulsby, Roberts, and Mitchener, 2000).

The concept of static yield stress is similar to the one already reported in the literature for different systems (Balmforth, Frigaard, and Ovarlez, 2014; Cheng, 1986; Coussot, 2014). While the fluidic yield stress differs from the idea of a dynamic yield stress presented in previous studies (Cheng, 1986; Toorman, 1997). The dynamic yield stress

is related to the value of the stress required to stop the flow of fluid. It is usually measured by performing a decreasing shear rate/stress sweep. This approach is suited to study the recovery of a structure that is broken at high shear. Fluidic yield stress represents the stress value, that is required for reaching the state when partially destructed structure of a sample is completely broken. On the other hand, the protocol proposed by Claeys et al. (2015) starts with a pre-shearing step at a very high shear rate (1000 s^{-1}), which is used to destroy all the structure

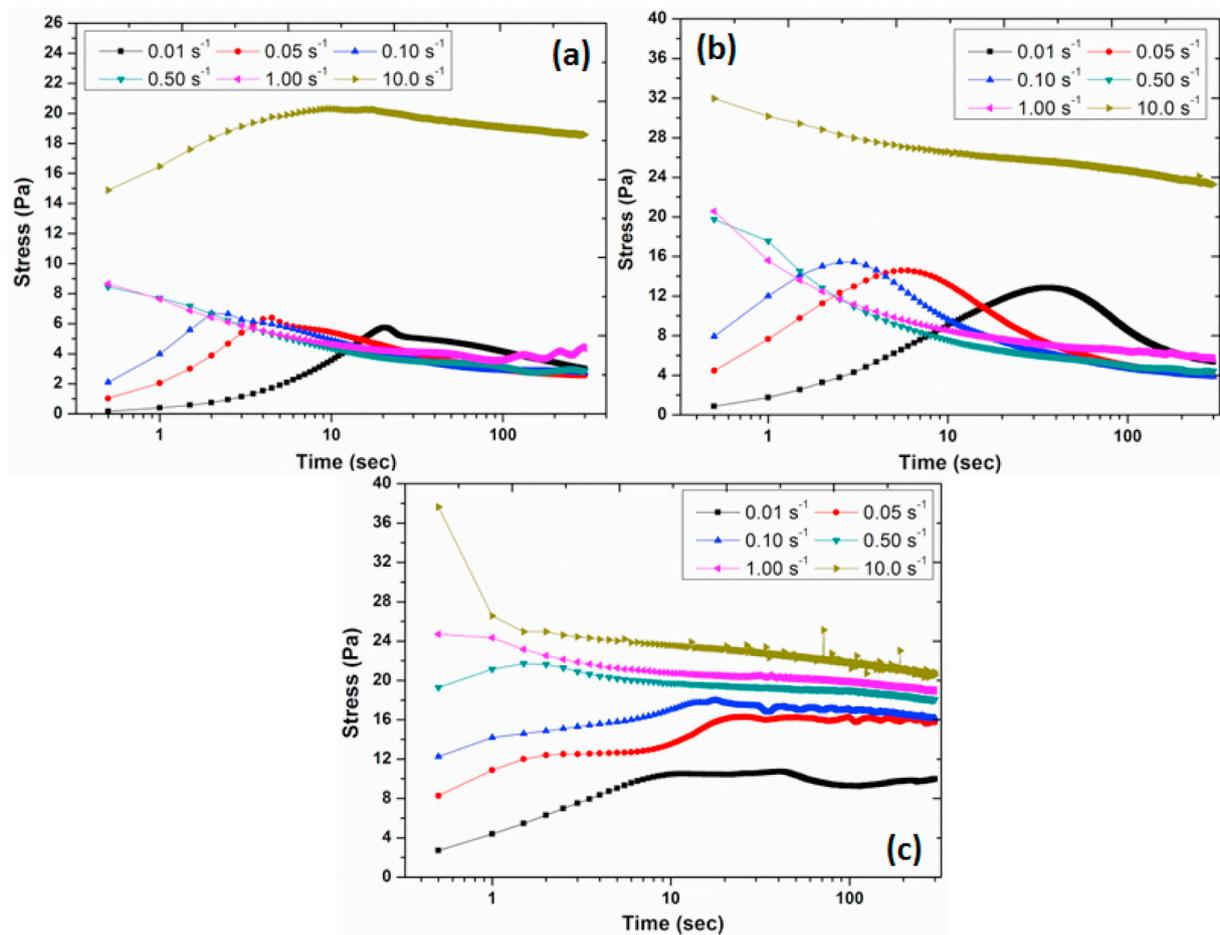


Fig. 6. Stress evolution as a function of time for (a) Couette, (b) parallel plate and (c) vane geometries at various shear rates for fluid mud layer. Solid line is just a guide for the eye.

within the sample. This protocol is, therefore, not suitable to measure the static yield stresses of the “undisturbed” mud samples. Furthermore, this protocol is based on nine cycles of applying/not applying shear and the total time of the experiment is about 20 min per sample. This protocol is therefore not suited to analyze the yield stresses of the large number of samples.

The outcome of stress sweep tests can also be presented in terms of strain as a function of shear stress on a double logarithmic plot, as shown in Fig. 3b. Two different slopes can be identified; the smaller slope is linked with the elastic deformation of the system while the viscous flow of the material is associated with the larger slope of the stress-strain curve. Two-step yielding was recognized based on the change in slope of stress-strain curves. Static and fluidic yield stress values can be determined from the transition between these two slopes by extending the straight lines from the regions below and above the yield point. A similar representation of the outcome of stress sweep tests was also reported for fresh cement mortars using vane type geometry (Qian and Kawashima, 2018). The determination of yield stress from viscosity curves is much easier than from the strain behavior due to the fact that the viscosity decline appear sharper to the eye as compared to the change in slope of the strain. Pre-consolidated (PS) and consolidated (CS) mud layers displayed similar trends of strain as a function of shear stress for different geometries (shown in Fig. S1b & d).

4.2. Oscillatory amplitude sweep

Preliminary oscillatory amplitude sweep tests were performed at different frequencies to determine the suitable frequency for a particular geometry (data not shown). It was observed that at higher

frequencies there was no cross-over between G' and G'' . This implies that the system retains its solid-like characteristics even at high amplitudes. This is, however, due to the fact that at these high frequencies the observation time was not long enough and, therefore, higher amplitudes would be required to destroy the structure of the system, i.e., to impart fluidization. A suitable frequency for all the geometries and mud layers was found to be 1 Hz. Similar results for oscillatory strain sweeps as a function of different frequencies for natural mud samples (Yueqing Bay, China) were also reported in the literature (Yang and Yu, 2018). They showed that the cross-over amplitude (i.e., $\delta = 45^\circ$) shifted towards higher values with the increase in frequency.

The results of oscillatory amplitude sweep tests performed at 1 Hz using different geometries are shown in Fig. 4. As already discussed, different criteria can be used to determine yield stress values from the output of amplitude sweep tests such as a sharp decline in storage modulus, cross-over between G' and G'' , and a decline in elastic stress. The storage and loss moduli of the fluid mud layer as a function of oscillation amplitude at a frequency of 1 Hz for different geometries is displayed in Fig. 4a. The static (first yield point) and fluidic (second yield point) yield stress values from this plot can be determined by the intersection of a line representing G' behavior below the yield points with the line demonstrating the behavior of G' above the yield points. This approach was previously reported to determine the yield stress values of foams, carbopol gels, emulsions and commercial lubricating greases (Cyriac, Lugt, and Bosman, 2015; Dinkgreve, Paredes, Denn, and Bonn, 2016; Rouyer, Cohen-Addad, and Höhler, 2005).

Two-step yielding was also observed in oscillatory amplitude sweep tests for all three considered geometries. Similar two-step yielding in oscillatory amplitude sweep tests was also reported in literature for

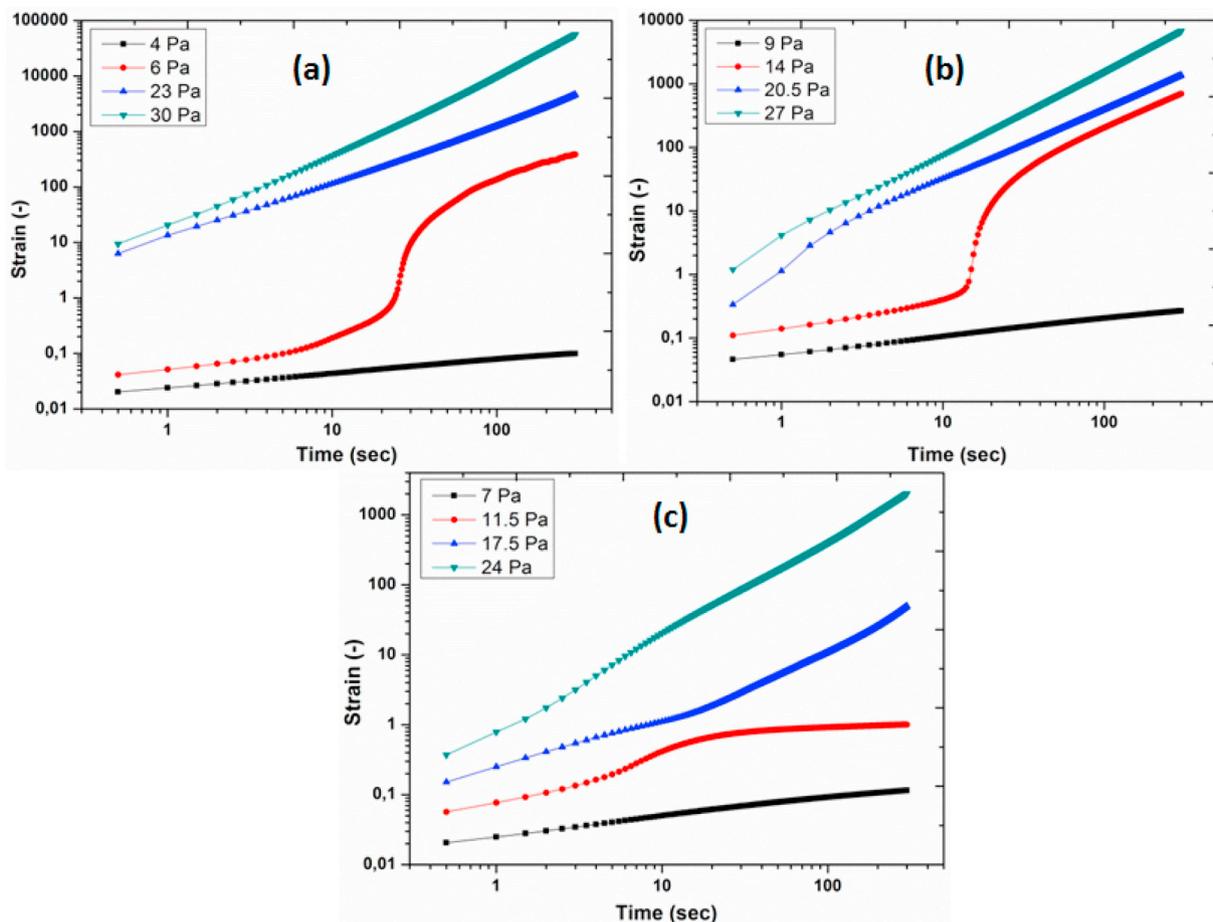


Fig. 7. Strain as a function of time for fluid mud layer using (a) Couette (b) parallel plate and (c) vane geometries at various applied stresses. Solid line is just a guide for the eye.

silica dispersions (Potanin, 2019), capillary suspensions (Ahuja and Gamonpilas, 2017), surfactant pastes (Shukla, Arnipally, Dagaonkar, and Joshi, 2015), etc. For example, for CoNi nanoplatelet based magneto-rheological fluids (MRFs), two-step yielding was observed in amplitude sweep tests which was attributed to the inter-cluster bond breaking (first yield point) and the cluster breaking (second yield point) (Arief and Mukhopadhyay, 2019). The strain hardening phenomenon was also observed for these MR fluids (increase in G' or G'' after the first yield point) (Arief and Mukhopadhyay, 2019). The similar increase in storage modulus, after the first yield point, was also observed for the mud samples, particularly for pre-consolidated and consolidated

sediments, which suggested the existence of strain hardening/jamming phenomenon in the mud samples. The possible mechanism of two-step yielding in mud samples is pictorially represented in Fig. 5.

Phase angle is plotted as a function of the oscillation amplitude for different geometries (Fig. 4b) to use the criterion of cross-over between G' and G'' (i.e., phase angle of 45°) for static yield stress measurements. The fluidic yield stress values were determined from the point where the phase angle started to increase again after a decline. This criterion of defining the fluidic yield stress was not very straightforward for the vane geometry, that is why not appropriate for this geometry. The oscillatory data is re-plotted in the form of the elastic stress (see Eq. (1))

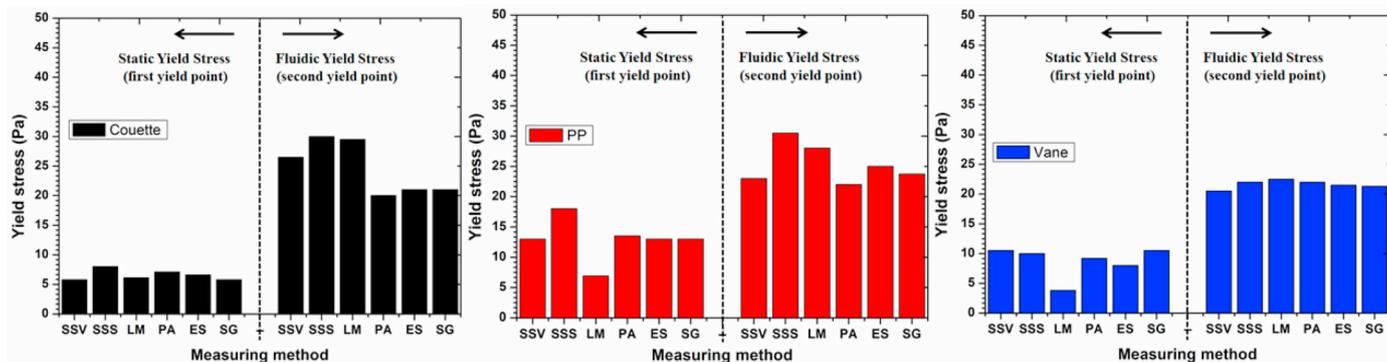


Fig. 8. Comparison of static and fluidic yield stress values of fluid mud layer using different geometries; SSV = viscosity decline from stress sweep, SSS = deformation slopes from stress sweep, LM = loss modulus decline from oscillatory amplitude sweep, PA = phase angle from oscillatory amplitude sweep, ES = elastic stress from oscillatory amplitude sweep, SG = stress growth.

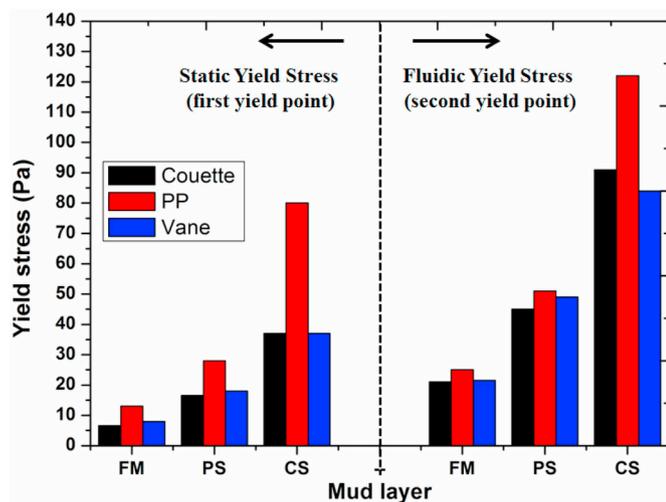


Fig. 9. Static and fluidic yield stress values for different mud layers using Couette, parallel plate and vane geometries obtained from decline in elastic stress.

as a function of amplitude for different geometries (Fig. 4c). Below the static yield stress value, the elastic stress showed a linear increase with the applied amplitude and a deviation from linearity was observed at static yield stress value of the sample. This approach was found to be appropriate for vane geometry as well to estimate the both static and fluidic yield stress values. This methodology was reported to successfully determine the yield stress values of cyclopentane hydrate slurry (Ahuja, Zyliftari, and Morris, 2015) and also the two-step yielding of cocoa suspensions in vegetable oil and water (Ahuja and Gamonpilas, 2017). Similar results of oscillatory measurements for pre-consolidated and consolidated mud layers were also observed, as shown in Figs. S2-S3.

4.3. Stress growth experiments

Stress growth experiments were carried out at shear rates ranging from 0.01 to 10 s⁻¹ using different geometries for mud samples. The results for fluid mud layer in terms of stress evolution as a function of time is shown in Fig. 6. As already explained in Sec. 2, different criteria can be used to determine the yield stress values from this method. In our case, peak stress value was chosen to represent the yield stress as it was easy to define. In case, where the peak stress was not observed, the equilibrium value of stress was used as a yield stress value (i.e., at higher shear rate). It can clearly be seen from Fig. 6a that at lower shear rates for Couette geometry, the peak stress corresponded to the static yield stress (first yield point) while the response at higher shear rate (10 s⁻¹) resulted in peak stress value similar to the fluidic yield stress (second yield point), as obtained through stress and amplitude sweeps.

Similarly, at lower shear rates for parallel plate geometry (Fig. 6b), the peak stress values were closer to the first yield point whereas for higher shear rates, the equilibrium stress value was considered and it was similar to the fluidic yield stress (second yield point). In contrast, for vane geometry (Fig. 6c), two-step yielding was evident for lower shear rates (0.01–0.10 s⁻¹) with two peak stress values. However, for

higher shear rates, one or no peak stress was observed due to the destruction of structure at such higher shear rates. The selection of the appropriate shear rate is critical for obtaining the desired yield stress from this method, as already explained in the literature (Rogers, Callaghan, Petekidis, and Vlassopoulos, 2010; Stokes and Telford, 2004; Yuan, Zhou, Khayat, Feys, and Shi, 2017). The similar approach was also reported in the literature to investigate the two-step yielding in colloidal glasses (Pham et al., 2008) and two peak yield stresses were observed as a function of applied strain. The similar results of stress growth tests were also observed for pre-consolidated and consolidated mud layers, as shown in Figs. S4-S5.

4.4. Creep tests

The creep tests were performed with the stress values taken from the four regions of viscosity curves obtained through the stress sweep tests: (i) stress below first yield point (first plateau), (ii) stress above first yield point, (iii) stress below second yield point (second plateau), and (iv) stress above second yield point. The resultant strain as a function of experimental time for fluid mud layer using different geometries is shown in Fig. 7. It is clearly evident from Fig. 7 that the creep responses of fluid mud sample for selected stresses were significantly different for all three geometries. In short, below the static yield stress (first yield point), the values of strain were too small and less dependent on experimental time. On the other hand, a sudden increase in strain was observed above the static yield stress which became again linear after 30 s, as a function of experimental time. The initial increase in strain can be attributed to the breakdown of inter-connected flocs while the linear behavior after 30 s can be linked to the existence of individual flocs, which resisted the applied stress.

A more or less linear increase in strain, with higher values, was observed for the stresses below the second yield point which was also evident for the stresses higher than the second yield point but with different slope. This difference in slopes can be associated with the breakage of flocs to the smaller flocs or individual particles. Hence, this approach also confirmed the existence of two-step yielding in mud sediments by having different levels of structural breakdown in creep tests. The creep method has been reported to successfully measure the yield stress values of fresh cement pastes, polymeric gels and emulsions using cone and plate and Couette geometries (Dinkgreve, Paredes, Denn, and Bonn, 2016; Qian and Kawashima, 2016). The creep tests have also been used in the literature to study the two-step yielding in dilute colloidal gels of polymethyl methacrylate (PMMA) microspheres (Chan and Mohraz, 2012). They also found different material's response at different applied stresses, due to the existence of two-step yielding in the investigated samples. Pre-consolidated and consolidated mud samples also displayed the similar response for creep test (Figs. S6-S7)

4.5. Comparison of yield stress values

The comparison of static and fluidic yield stress values, obtained from different geometries using various rheological methods for fluid mud layer is shown in Fig. 8. It is clear from the figure that both static and fluidic yield stress values, obtained through different geometries and rheological methods, were in more or less same order of magnitude. Furthermore, it can be easily seen that the static yield values were

Table 3
Pros and cons of different geometries used for the rheological analysis of mud samples.

| Geometry | Analyzing two-step yielding (static and fluidic) | Analyzing absolute yield stress values (no extensive disturbance at the onset of the experiment) | Analyzing solid-like samples |
|----------|--|--|------------------------------|
| Couette | X | | |
| PP | X | X | X |
| Vane | X | X | X |

Table 4
Pros and cons of different rheological methods used for the analysis of mud samples.

| Rheological method | Pros | Cons |
|-----------------------------|--|---|
| Stress sweep | Fast for analyzing all structural break-downs. | Analyzing the results can lead to different yield stress values depending on the accuracy of the extrapolation. |
| Oscillatory amplitude sweep | Easy to define the yield stress values from cross-over between G' and G'' and the decline of elastic stresses. | Analyzing the moduli decline can lead to different yield stress values depending on the accuracy of the extrapolation. Phase angle criterion is not appropriate to define static yield stress because the structural breakdown can happen before the cross-over. |
| Stress growth | Fast | Highly dependent on the applied shear rates. Different yield stress values can be obtained depending on the criterion used to define the yield stress. |
| Creep | Suitable for verifying the yield stress values obtained through stress sweep tests and also two-step yielding. | Prior knowledge of yield stress values is required. Time consuming because of the need to perform series of experiments. The range of yield stresses can be estimated, instead of a single value. |

Table 5
Comparison of yield stresses of mud samples from literature with this study.

| Study Area | Density Range (kg m^{-3}) | Fluidic/Bingham Yield Stress Range (Pa) | Ref. |
|------------------------------------|--------------------------------------|---|---|
| Port of Rotterdam, the Netherlands | 1168 ^a | 7 | (Van Kessel and Blom, 1998) |
| Eckernförde Bay, Germany | 1038–1280 | 1.07–20.50 | (Fass and Wartel, 2006) |
| Hangzhou Bay, China | 1145–1634 | 0.55–40 | (Huang and Aode, 2009) |
| Mouth of Yangtze River, China | 1650–1700 | 910–2810 | (Yang, Yu, Tan, and Wang, 2014b) |
| Shoal of Hangzhou Bay, China | 1705–1741 | 772–2140 | (Yang, Yu, Tan, and Wang, 2014b) |
| Yangcheng Lake, China | 1651–1691 | 2070–3960 | (Yang, Yu, Tan, and Wang, 2014b) |
| Lianyungang Port, China | 1098–1305 | 0.098–28.029 | (Xu and Huhe, 2016) |
| Port of Santos, Brazil | 1085–1206 | 5–334 | (Fonseca, Marroig, Carneiro, Gallo, and Vinzón, 2019) |
| Port of Rio Grande, Brazil | 1132–1308 | 5–350 | (Fonseca, Marroig, Carneiro, Gallo, and Vinzón, 2019) |
| Port of Itajaí, Brazil | 1138–1360 | 5–299 | (Fonseca, Marroig, Carneiro, Gallo, and Vinzón, 2019) |
| Amazon South Channel | 1293–1512 | 5–379 | (Fonseca, Marroig, Carneiro, Gallo, and Vinzón, 2019) |
| Lianyungang Port, China | 1107–1546 | 0.014–380 | (Nie, Jiang, Cui, and Zhang, 2020) |
| Port of Hamburg, Germany | 1087–1484 | 2.44–312 | (Shakeel et al., 2020) |
| Port of Hamburg, Germany | 1134–1186 | 20–210 | This study |

^a Calculated from mud concentration.

comparatively lower (i.e., 5.8–7.1 Pa) for the Couette geometry while the fluidic yield stress values were smaller (i.e., 20.5–22.5 Pa) for the vane geometry. This result shows that the Couette geometry is more effective in breaking the network of flocs/aggregates (first yield point) while for the breakdown of flocs into individual particles (second yield point), vane geometry is better. Due to this effective bulk structural breakdown with vane geometry, it has been utilized as a mixer for preparing polymeric blends with enhanced mechanical and structural properties (Qu et al., 2013; Xiaochun, Zhongwei, Guangjian, Zhitao, and Baiping, 2015).

Moreover, static yield stress values were higher for parallel plate geometry (i.e., 6.9–18 Pa) than the Couette geometry (i.e., 5.8–7.1 Pa). This may be due to the fact that parallel plate geometry offered less disturbance to the samples when the top plate is put into position. When the bob is put into place, the sample is clearly more disturbed. The influence of this disturbance on the fluidic yield stress values was almost negligible as similar values were observed for both geometries. Similar trends in static and fluidic yield stress values, obtained from different geometries using several rheological methods, were also observed for the other two mud layers (see Figs. S8–S9).

Fig. 9 shows the values of static and fluidic yield stresses obtained by elastic stress approach for different mud layers. Elastic stress approach is very straightforward for all the geometries to identify the yield points, that is why, this approach was selected to compare the two-step yielding behavior of different mud layers. The sample having the highest density values (i.e., 1186 kg m^{-3}) displayed the large yield stress values (static yield stress = 37–80 Pa; fluidic yield stress = 84–122 Pa) due to their higher solids volume fraction. The pros and cons of the investigated geometries and rheological methods for the analysis of two-step yielding in mud sediments are summarized in Tables 3 and 4. The values of the yield stresses obtained in the present study were compared with the yield stress values of the mud

samples reported in literature (Table 5). The comparison showed that the values of yield stresses for mud samples obtained from the Port of Rotterdam, the Port of Santos, Lianyungang Port, and the Port of Hamburg, are similar (within the similar density ranges). The optical micrographs of diluted mud samples is shown in Fig. S10, which verified the presence of inter-connected network of flocs.

Stress sweep tests with Couette geometry were proven to be practical and time efficient tests for measuring all structural breakdowns within the mud samples associated to static and fluidic yield stresses (two-step yielding). The fluidic yield stress value is an important parameter to define a limit for the nautical bottom in ports and waterways. Fluidic yield stress was linked with the complete structural breakdown in fluid mud which is needed for controllability and maneuverability of vessels. Static yield stress of fluid mud can be a suitable parameter for characterizing the mud in the small energy regions of the harbor.

5. Conclusion

In this study, a detailed and systematic rheological analysis of natural mud samples from the Port of Hamburg (Germany) was performed using different geometries and rheological methods. Three mud layers, fluid mud, pre-consolidated and consolidated mud (FM, PS and CS) were chosen for analysis having densities ranging from 1134 to 1186 kg m^{-3} , by using different measuring geometries. Concentric cylinder (Couette), parallel plate, and vane geometries, were used to perform rheological experiments such as stress sweep, oscillatory amplitude sweep, creep, and stress growth.

Two-step yielding behavior was observed for the mud samples in stress sweep and amplitude sweep tests. The first yield point (static) was linked with the breakage of interconnected network of aggregates/flocs while the second one (fluidic) was attributed to the collapse of

aggregates into the smaller flocs or individual particles. The suggested possible mechanism for two-step yielding (breaking of flocs) is a hypothesis based on our literature review. Further research is required to understand the origin of this two-step yielding. Creep test is not a straightforward method for analyzing the yield stress values of samples as in particular one must have a prior knowledge of the stress range in which the yielding occurs. Vane geometry is to be used in case of very consolidated systems (solid-like), where Couette geometry cannot because the bob can get stuck during the experiment. The static yield values were comparatively lower (i.e., 5.8–7.1 Pa) for the Couette geometry while the fluidic yield stress values were smaller (i.e., 20.5–22.5 Pa) for the vane geometry. This result shows that the Couette geometry is more effective in breaking the network of flocs/aggregates (first yield point) while for the breakdown of flocs into individual particles (second yield point), vane geometry is better. Parallel plate geometry is not a suitable option for investigating liquid-like samples because the sample can spread out of the gap during the shearing action. Parallel plate geometry can, however, be a good option to measure the absolute values of yield stresses because it offers less disturbance in the sample structure when the top plate is set into place prior to the experiment. Our study showed that Couette and parallel plate geometries are the most suitable geometries for analyzing all the structural breakdowns of the samples. Cone and plate geometry is not designed for mud sediments having large particles because of the very narrow gap between the cone and plate.

Besides mud sediments, the present comparison study can also be of use to the researchers in assessing the applicability of studied geometries and rheological methods for their own system under investigation, particularly system with two-step yielding behavior. This can be a suspension, an emulsion or a gel, and be linked to a different domain of application. Furthermore, as discussed in Sec. 3.1, the physical properties of muddy sediments are strongly affected by the presence of small amounts of organic matter. Hence, this study provides a step forward for investigating the effect of organic matter on the rheological properties of marine sediments by efficiently estimating the rheological fingerprint of mud sediments.

Declaration of Competing Interest

There are no conflicts of interest to declare.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.margeo.2020.106247>.

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