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Revising the definition of fluid mud by establishing new protocols for rheological measurements

Réviser la définition de la crème de vase en établissant de nouveaux protocoles pour les mesures rhéologiques

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ABSTRACT: Fluid mud can be described as a mixture of water, organic matter and mainly cohesive mineral sediment that is usually found in estuaries and in rivers with low-intensity currents. It is in a transient state and can densify over time unless mixing (produced by mechanical and/or natural forces) prevents its gelling. Typically, fluid mud exhibits bulk densities between 1080 and 1200 kg/m^3. It has a weak strength that, in low energetic conditions, develops over time forming a structured bed of considerably higher rigidity. The current definitions of fluid mud are based on the density of the sediment. These definitions are ambiguous since the proposed critical parameters do not account for the possible strength developments in fluid mud. We propose to use rheological properties for distinguishing the fluid mud layer from the suspension layer above and the consolidated mud layer underneath. The protocols involve the use of a rotational bob (Couette) rheometer that can perform oscillations, shear stress and shear rate controlled experiments. A number of natural mud samples were used for testing the protocols.

RÉSUMÉ: La crème de vase peut être décrite comme un mélange d'eau, de matière organique et principalement de sédiments minéraux cohésifs que l'on trouve généralement dans les estuaires et dans les rivières avec des courants de faible intensité. Elle est dans un état transitoire et peut se densifier avec le temps, à moins qu'elle ne soit mélangée (par l'action de forces mécaniques et/ou naturelles) ce qui l'empêcherait de se gélifier. En règle générale, la crème de vase présente des densités apparentes comprises entre 1080 et 1200 kg/m^3. Elle a une faible contrainte seuil qui se développe avec le temps, formant un lit structuré de rigidité considérablement plus élevée. Les définitions actuelles de la crème de vase se basent sur la densité du sédiment. Ces définitions sont ambiguës car les paramètres critiques proposés prennent pas en compte les possibles développements de contraintes dans la vase,. Nous proposons d'utiliser les propriétés rhéologiques pour distinguer la couche de crème de vase de la couche en suspension qui se trouve au-dessus et de la couche de vase consolidée qui se trouve en-dessous. Les protocoles nécessitent l'usage d'une rhéomètre type Couette pouvant effectuer des mesures en mode oscillations, des mesures de contrainte et taux de cisaillement contrôlés. Un certain nombre d'échantillons de vase naturelle ont été utilisés pour tester les protocoles.

Keywords: Yield stress; Fluid mud; Rheological measurements; Protocols

1 INTRODUCTION

Fluid mud typically consists of water, mineral particles and organic matter (such as microbial slime). This organic matter can be represented as a network of polymer chains in which mineral particles can be trapped. Hydrodynamic conditions ensures that fluid mud can be seen as a suspension or a single visco-elastic medium.

The change from two-phase fluid to gel of what is commonly called “fluid” mud has led to a variety of definitions for this medium. Fluid mud has been defined as “a suspension of cohesive sediments having concentrations above the gel point, i.e., 10-100 g/L, and behaving like a non-Newtonian fluid” by Winterwerp et al. (Winterwerp and Van Kesteren 2004). McAnally & coworkers define fluid mud as: “a concentrated suspension of fine-grained sediments in which settling is considerably hindered by the presence of flocs, but at the same time absence of strong interconnected bonds which can prevent mobility" (McAnally et al. 2007). Implicitely, these definitions rely on external energy sources, such as pressure gradients or waves, to keep the mud in a fluidic state. In low energetic environments, however, fluid mud can gelify and at longer times consolidate. It is important to note that this gel state is reversible and that fluid mud will liquefy again in favorouble hydrodynamic conditions. This results in the large variation of properties (dry density and mechanical properties) over mud layer thicknesses and time that can be observed in natural systems.

The presence of a complex visco-elastic fluid mud layer over the sea or river-bed can make the navigation in ports and waterways quite challenging due to the following reasons (Kirichek et al. 2018):

- Traditional acoustic methods are not reliable to detect fluid mud layers because of the strong gradients in (fluid) mud properties that render the interpretation of acoustic data ambigious.
- Another hurdle for navigation in fluid mud areas is the generation of internal waves (undulations). These undulations can affect the controllability and manoeuvrability of a vessel for ship navigation in the vicinity of water-mud interface.

At present, the following definition of the nautical bottom is widely accepted for navigational purposes in fluid mud areas: "The nautical bottom is the level where physical characteristics of the bottom reach a critical limit beyond which contact with a ship’s keel causes either damage or unacceptable effects on controllability and manoeuvrability" (PIANC 2014). Most Ports, for the sake of practicability, have adopted fluid mud density as a creteria to estimate an acceptable nautical bottom. However, the Port of Emden, has adopted yield stress as the criteria for defining nautical bottom since 2005 (Wurpts 2005). Port of Emden adopted a yield stress of 100 Pa as a criteria, above which fluid mud is navigable. This yield stress is port specific and depending on the dredging method, ship navigation and the properties of organic matter in this port.

Rheological properties, particularly yield stress, can be determined in the laboratory by using either the stress controlled or rate controlled mode of the rheometer. Different geometries are also available to choose depending upon the consistency of the sample under investigation. In literature, several methods are also reported to measure the yield stress of complex systems including suspensions and gels (Dinkgreve et al. 2016, Rahman et al. 2017, Claeys et al. 2015). Due to the complex behavior of fluid mud there is a need to develop a specific practical protocol using a suitable geometry for measuring yield stress values of mud suspensions. The overall objective of this study is to compare different rheological methods using different geometries for yield stress measurements of mud suspensions, particulay fluid mud, and from this study finding the most appropriate method and geometry.

2 MATERIALS AND METHODS

2.1 Materials

In this study, natural mud samples were taken from the Port of Hamburg, Germany. The collected samples were divided into three layers, i.e., fluid mud (FM), pre-consolidated (PS) and consolidated (CS), based on the differences in their visual consistency (Fig. 1). The samples were packed in a sealed container and shipped to the laboratory. The median particle size (D_{50}) of fluid mud, pre-consolidated and consolidated mud layers was 16.7, 17.0 and 16.9 µm, respectively. Particle size distribution of these three layers, measured by static light scattering technique, is shown in Fig. 2. All the rheological experiments were performed after the homogenization of the samples.

2.2 Equipment

A HAAKE MARS I rheometer (Thermo Scientific, Karlsruhe, Germany) was used to perform the rheological measurements. This rheometer can either be operated in stress controlled, 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 flow curve analysis.

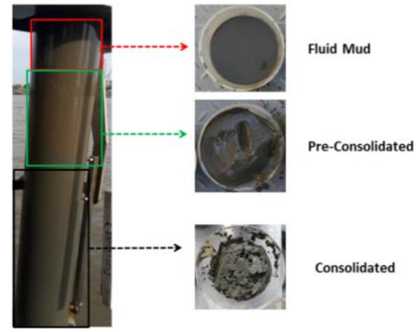

Figure 1. Different mud layers at the Port of Hamburg

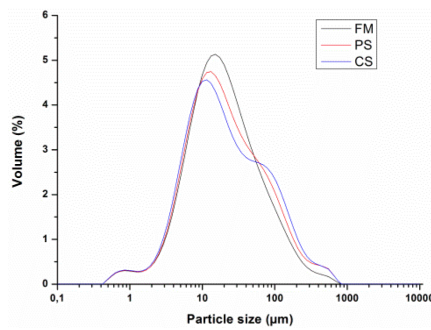

Figure 2. Particle size distribution of fluid mud (FM), pre-consolidated (PS) and consolidated (CS) samples

Five different geometries, including concentric cylinder (Couette), cone and plate (CP), smooth (PP) and roughened parallel plates (PP-S), and vane geometries were used to perform different tests. For concentric cylinder geometry, the cup inner and bob outer diameters were 27 mm and 25 mm, respectively and the distance from the bottom of the cup was 5.30 mm. In case of vane geometry, the cup inner and vane outer diameter were 27 mm and 22 mm, respectively and the distance from the bottom of the cup was 1 mm. Cone and plate geometry having diameter of 60 mm with 2^{o} cone angle and 0.104 mm gap between the cone and plate was used to perform experiments. The diameter of smooth and roughened parallel plates was 35 mm and 2 mm gap was used between the plates for rheological experiments.

2.3 Methods

2.3.1 Stress Sweep

Stress sweep tests for the all geometries were performed using controlled stress mode of the rheometer. A stress ramp of 0.2 Pa/s was applied and the stress was increased from 0 to 500 Pa, depending upon the consistency of the sample. The corresponding torque was measured, and the shear rate and viscosity were then determined.

2.3.2 Amplitude Sweep

Preliminary amplitude sweep tests at different frequencies were also performed to analyze the suitable value of frequency for amplitude sweep tests (data not presented here). The oscillatory amplitude sweep tests were carried out by increasing shear stress from 0 to 100 Pa, depending upon the consistency of the samples, at a constant frequency of 1 Hz. The material's out-of-phase response was obtained in terms of G' (real) and G'' (imaginary) parts of the complex modulus G^* as a function of the applied amplitude. The complex modulus, G^* and phase angle, δ can then be calculated as follows:

$$G^* = \sqrt{G'^2 + G''^2} \qquad (1)$$

$$\delta = tan^{-1}\frac{G''}{G'} \qquad (2)$$

The stress at which a sharp decrease in complex modulus is observed or at which G' crosses G'' can be taken as the yield stress (Perge et al. 2014, De Graef et al. 2011).

2.3.3 Creep

In the creep experiment, a constant stress, τ_0 was applied to the sample and the resultant deformation, $\gamma(t)$ was recorded as a function of time. The first applied stress is chosen to be below the expected yield stress, as determine by other experiments. The applied stress is then increased. The creep compliance $J(t)$ was then determined as follows:

$$J(t) = \frac{\gamma(t)}{\tau_0} \qquad (3)$$

Below the yield point, the applied stress can lead towards a constant value of compliance after some time. Above yielding, a sharp increase in the creep compliance, as a function of time, can be observed.

2.3.4 Flow Curve

The flow curve analysis was performed using controlled rate mode of rheometer by carrying out shear rate ramp from 0 to 300 s^{-1} without giving enough time between each point of measurement to attain equilibrium values.

3 RESULTS AND DISCUSSION

The apparent viscosity of fluid mud layer as a function of shear stress for different geometries is shown in Fig. 3. The viscosities obtained from Couette and parallel plate geometries display two characteristic plateaus followed by two declines. The stress values associated with these two declines can be referred to as static yield stress, τ_y^s (first decline) and fluidic yield stress, τ_y^f (second decline), which are related to the particular level of structural breakdown, as shown in Fig. 3.

These two characteristic viscosity plateaus were not observed in case of roughened parallel plate and vane geometries. This may be linked to the fact that these geometries enables the fluidization of the whole sample even at lower shear stresses. Cone and plate geometry was discarted for further experimentation because the response of the material was very scattered due to the presence of bigger particles within the small gap of cone and plate.

The complex modulus of fluid mud layer as a function of oscillation amplitude at a frequency of 1 Hz for different geometries is presented in Fig. 4. The stress value where a decrease in complex modulus was observed can be seen as a yield stress.

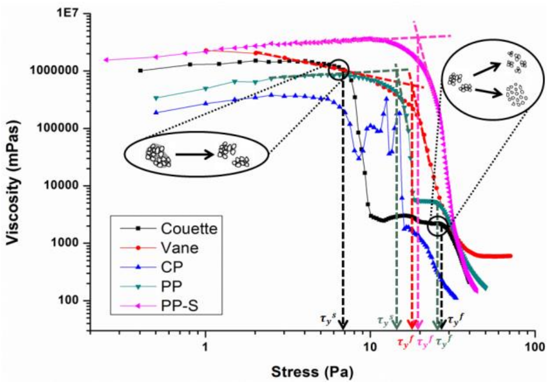

Figure 3. Apparent viscosity as a function of shear stress for fluid mud layer using different geometries

Phase angle was also plotted as a function of oscillation amplitude for different geometries (Fig. 5). The cross-over between G' and G'' (i.e., $\delta = 45°$) can also considered as a measure of yield stress. However, this criterion of yield stress measurement is not very appropriate because some structure was already broken down before the cross-over of moduli. Moreover, vane and roughened parallel plate geometries gave higher values of yield stresses from cross-over, which again confirms the inherent nature of these geomerties to distrupt the whole sample and, that is why, these yield stress values were stated as fluidic yield stresses for these two geometries.

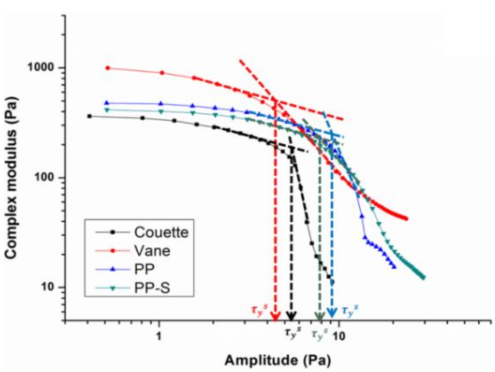

Figure 4. Complex modulus as a function of oscillation amplitude for fluid mud layer using different geometries

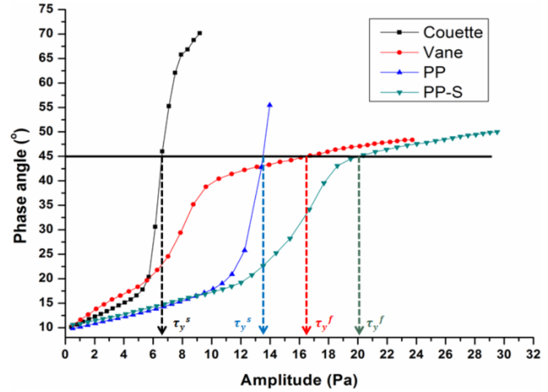

Figure 5. Phase angle as a function of oscillation amplitude for fluid mud layer using different geometries

The flow curves of fluid mud layer are shown in Fig. 6 for different geometries. At lower shear rates, the response of the material was very scattered which may be due to the sensitivity of the equipment at such lower shear rates. Couette and parallel plate geometries showed a minimum in flow curve which was absent in case of vane and roughened parallel plate geometries. This absence of minimum can again be related to the inherent nature of these geometries to destroy the sample‘s structure at lower stresses/shear rates. This fact needs further investigation by changing the experimentation protocol to allow more time during each point of measurement.

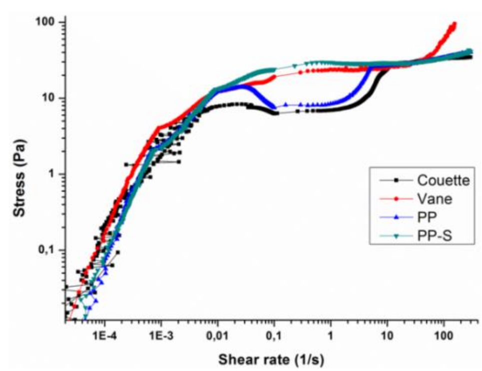

Figure 6. Flow curves of fluid mud layer using different geometries

The minimum in flow curve is more clearly displayed in Fig. 7 by concentrating on the appropriate range of shear rate. Static yield stresses were estimated, for all the geometries, from the y-intercept of the flow curves presented in Fig. 7. Fluidic yield stresses were also recorded for Couette and parallel plate geometries from the flow curve where a linear behavior started again after the minimum.

The creep tests were performed for mud suspensions at stress values taken from the stress sweep tests (i.e., static yield stress), as a starting point. The creep compliance of fluid mud layer for couette geometry is shown in Fig. 8. Below yield stress (i.e., 4 Pa), the material‘s response increased linearly and then became more or less constant as a function of time whereas a sudden increase in compliance was observed at/above yield stress (i.e., 5 Pa) .

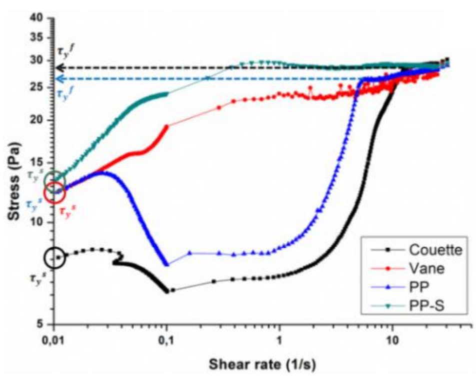

Figure 7. Flow curves of fluid mud layer for selected range of shear rate using different geometries

The creep compliance of fluid mud layer, above yield point, for different geometries is presented in Fig. 9. Couette and parallel plate geometries showed a sharp increase in creep compliance above yield stress, whereas in case of vane and roughened parallel plate geometries the deviation was ambiguous. This fact is in accordance with the previous result of stress sweep tests that these two geometries (vane and roughened parallel plate) enable the rapid fluidization of the whole sample.

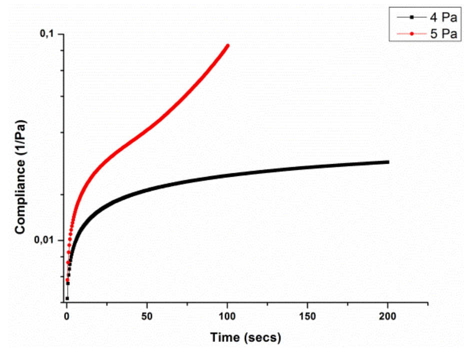

Figure 8. Creep compliance as a function of time for fluid mud layer using Couette geometry

Figs. 10-12 present the comparison of yield stress values of different geometries and different rheological methods for three mud layers. Static and fluidic yield stress values from stress sweep tests and flow curve analysis were in accordance with each other for all three mud layers. The fluidic yield stress values are very important as they can be used to define a limit for the nautical bottom in ports and waterways. The fluidic yield stress is linked with the structural breakdown in fluid mud which is needed for controllability and manoeuvrability of vessels. The yield stress values obtained from moduli decline of amplitude sweep tests were lowest for all the geometries, which may be attributed to the fact that structural breakdown is much easier to attain by having oscillatory shear.

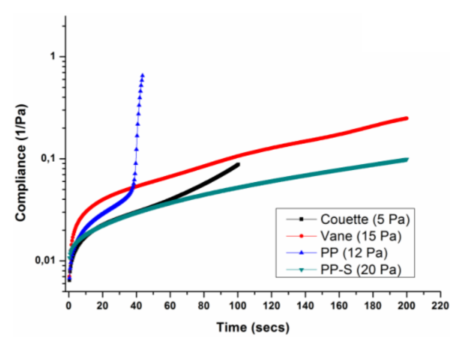

Figure 9. Creep compliance as a function of time for fluid mud layer above yield point using different geometries

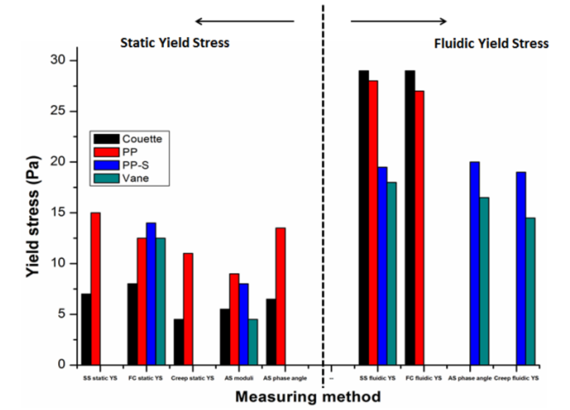

Figure 10. Comparison of yield stress values of fluid mud layer

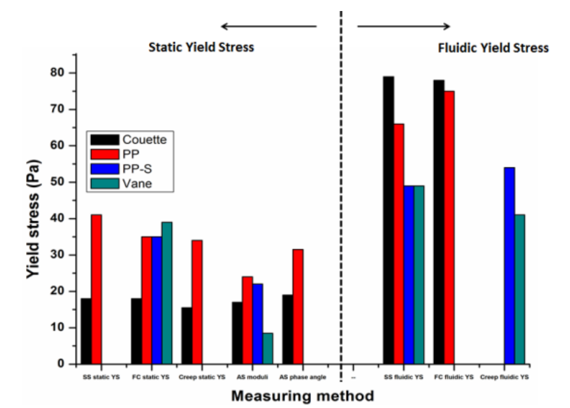

Figure 11. Comparison of yield stress values of pre-consolidated layer

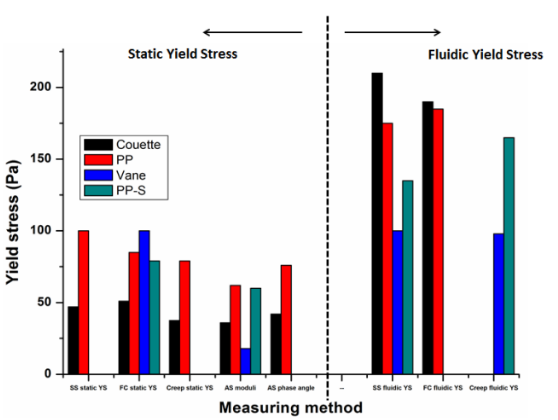

Figure 12. Comparison of yield stress values of consolidated layer

4 CONCLUSIONS

In this study, a detailed rheological analysis of natural mud samples using different geometries and rheological methods is presented. Three different mud layers having different consistencies were collected in the Port of Hamburg, Germany for anlysis. Five different geometries including concentric cylinder (Couette), cone and plate, smooth and roughened parallel plates, and vane geometries were used to perform rheological experiments such as stress sweep, amplitude sweep, creep and flow curves.

Stress sweep tests were proved to be practical, time efficient, and reliable tests for measuring the yield stress of mud suspensions. Amplitude sweep tests also offered very small values of yield stresses which can be interesting for practical applications. At very small shear rates in flow curves, the response of the material was very scattered due to the sensitivity of the equipment. A minima in flow curve was absent for vane and roughened parallel plate geometries which needs to be further investigated. Fluidic yield stresse values obtained from stress sweep tests and flow curves are can be potentially used for navigational purposes in ports and waterways.

Creep test is not a straight forward test to analyse the yield stress value of samples. Instead, one must have a prior knowledge of approximate range of yield stress values. A sharp deviation in the compliance curve was not observed in some cases i.e., for vane and roughened parallel plate geometries. Therefore, these geometries are not suitable to perform creep tests for yield stress measurement.

Vane geometry can be appropriate to use in case of very consolidated systems, while Couette geometry cannot be used for very consolidated systems because the geometry could stuck during the experiment due to the requirement of very high torque (shear stresses). Parallel plate geometry is not a suitable option to use for very liquid-like samples because the solid particles can splash out of the geometry during shearing action. Parallel plate geometry can be a good option to measure the absolute values of yield stresses because it offers less disturbance in samples while approaching the measurment position before experiment. As for comparing the rheological properties of different samples Couette geometry is also suitable.

Our study showed that Couette and parallel plate geometries are the most suitable geometries for analysing the two yield stresses of the samples. Cone and plate geometry is not suitable for the rheological analysis of mud suspensions because of the very narrow gap between the cone and plate.

The presented rheological analysis and protocols can be further used for characterizing mud samples in ports and waterways. This study can also help in revising the criteria for nautical bottom definition in ports by measuring the yield stress values in an appropriate manner and by differentiating different mud layers in the ports and waterways.

5 ACKNOWLEDGEMENTS

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