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Is density enough to predict the rheology of natural sediments?

Ahmad Shakeel[1,2] · Alex Kirichek[1,3] · Claire Chassagne[1]

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Abstract

Mud is a cohesive material which contains predominantly clay minerals, water, organic matter and some amounts of silt and sand. Mud samples can have complex rheological behaviour, displaying viscoelasticity, shear-thinning, thixotropy and yield stress. In this study, influence of organic matter on the rheological behaviour of different mud samples having similar densities is investigated. Four samples, collected from different locations and depths of Port of Hamburg (Germany) were selected. Two samples with the density of about 1210 kg/m^3 and two samples with the density of about 1090 kg/m^3 were analysed by different rheological tests, including stress ramp-up tests, flow curves, thixotropic tests, oscillatory amplitude and frequency sweep tests. Two yield stress regions (with two yield stress values stated as "static" and "fluidic" yield stresses) were identified for all the samples, and these regions, corresponding to a structural change of the samples were significantly different from sample to sample due to the differences in organic matter content. For lower density samples, the ratio of fluidic to static yield stress increased from 3 to 4.4 while it increased from 4.4 to 5.2 in case of higher density samples, by increasing the organic matter content. The thixotropic studies showed that the mud samples having lowest organic matter content (VH and KBZ) exhibit a combination of thixotropic and anti-thixotropic behaviours. The results of frequency sweep tests revealed the solid-like character of the mud within the linear viscoelastic regime. Mud samples having higher organic matter content (RV and RT) had a higher complex modulus (417 Pa and 7909 Pa) than the ones with lower organic matter content (13 Pa and 1774 Pa), for a given density. This study demonstrated that the density only is not a sufficient criterion to predict the rheology of different mud. Furthermore, even small amounts of organic matter content change significantly the mud rheological behaviour.

Keywords Natural sediments · Rheology · Yield stress · Density · Organic matter · Thixotropy · Fluid mud

Introduction

Mud, found in lake beds, river beds, or coastal seabed, is a cohesive material which contains predominantly clay minerals, water, organic content and some amounts of silt and sand. Estuarine mud beds are continuously disturbed by wave motion leading to the formation of different mud layers which can be classified as suspended mud, fluid mud, pre-consolidated/stationary mud and consolidated cohesive bed (Ross and Mehta 1989; Mehta 2013). Fluid mud is usually defined as a fluid having a density within the range of 1030–1300 kg/m^3 and concentrations about 10–100 g/L, in which settling is considerably hindered by the presence of flocs (Inglis and Allen 1957; Whitehouse et al. 2000; McAnally et al. 2007). Extensive research has been done on the floc size measurements in the laboratory for muddy sediments (Manning and Dyer 2002; Gratiot and Manning 2004; Manning et al. 2007; Spencer et al. 2010). The hindered settling behaviour was also observed for the sand/mud mixtures (Whitehouse and Manning 2007; Manning et al. 2010; Manning et al. 2011; Spearman et al. 2011; Spearman and Manning 2017). The presence of fluid mud makes the navigation in ports and waterways and the maintenance of dredging channels more difficult (May 1973; Parker and Kirby 1982; Kirichek et al.

✉ Ahmad Shakeel
A.Shakeel@tudelft.nl

Alex Kirichek
Alex.Kirichek@deltares.nl

Claire Chassagne
C.Chassagne@tudelft.nl

1 Faculty of Civil Engineering and Geosciences, Department of Hydraulic Engineering, Delft University of Technology, Stevinweg 1, 2628, CN Delft, The Netherlands

2 Department of Chemical, Polymer & Composite Materials Engineering, University of Engineering & Technology, KSK Campus, Lahore 54890, Pakistan

3 Deltares, Boussinesqweg 1, 2629, HV Delft, The Netherlands

2018). Mud samples are known to exhibit complex rheological behaviours like viscoelasticity, shear-thinning, thixotropy and yield stress (Coussot 1997; Van Kessel and Blom 1998). The presence of yield stress in mud results from the presence of aggregate networks, for particles volume concentration above 8% (Kranenburg 1994).

In literature, correlation between density or volume fraction of natural sediments and the yield stresses has been extensively reported. Xu and Huhe presented the rheological studies of natural mud at Lianyungang, China using a RS 6000 rheometer with the help of both steady and dynamic measurements (Xu and Huhe 2016). They correlated the yield stress values with the mud volume concentration and fitted the experimental data with an exponential empirical relation. Soltanpour and Samsami compared the rheology of kaolinite and Hendijan mud, northwest part of Persian Gulf (Soltanpour and Samsami 2011). They linked the rheological parameters with the water content in the natural and artificial sediments using exponential relations. Similarly, the relation between Bingham yield stress and the density of the natural mud sediments was also reported in the literature using empirical exponential relations (Carneiro et al. 2017; Fonseca et al. 2019).

As it is already reported in the literature, the cohesion and rheological properties of muddy sediments are strongly dependent on the presence of organic matter/biopolymer (Paterson et al. 1990; Paterson and Hagerthey 2001; Tolhurst et al. 2002; Wurpts 2005; Malarkey et al. 2015; Schindler et al. 2015; Parsons et al. 2016). However, a systematic analysis of the influence of organic matter on the rheological properties of mud samples and a quantification thereof is still missing. Therefore, in this study, we aim to answer the question “is the density or volume fraction of solids enough to predict/link the rheology of the natural sediments?” by studying the rheological behaviour of mud samples with similar densities, but different organic matter content. Four samples, collected from different locations at different state of consolidation from Port of Hamburg (Germany) were selected. Two of the samples had a similar density of about 1210 kg/m^3, whereas the two others had a similar density of about 1090 kg/m^3. Rheological analysis was performed by conducting stress ramp-up tests, flow curves, thixotropic tests, oscillatory amplitude and frequency sweep tests.

Experimental

In this study, the mud samples were collected from different locations (Vorhafen (VH), Köhlbrand (KBZ), Rethe (RT) and Reiherstieg Vorhafen (RV)) of Port of Hamburg, Germany using a 1-m core sampler (Fig. 1b). Figure 1a shows the selected locations at the port. These locations were chosen on the basis of a preliminary analysis, which showed that the selected locations have different amounts of organic matter content with significantly different rheological fingerprint. The collected samples were then divided into different layers based on the differences in their visual consistency such as fluid mud (FM), pre-consolidated (PS), pre-consolidated to consolidated (PS/CS) and consolidated (CS) sediments (Fig. 1c). Four different samples were then selected having similar densities, but with different organic matter content. The samples were packed in sealed containers and transported to the laboratory. The dry density of the minerals was assumed to be 2650 kg/m^3 (Coussot 1997). The bulk density of the mud samples was determined by the method reported in (Coussot 2017). Particle size distributions within the different mud layers were measured using static light scattering (Malvern MasterSizer 2000MU). There are several limitations of this instrument which need to be considered such as (i) it is based on the Mie theory which basically assumes the spherical shape of all the particles which can be inappropriate for mud sediments; (ii) only a certain concentration range is practically possible to measure, if it is too low or too high, a warning message will appear; and (iii) for samples with higher sand fractions, this techniques gives higher average particle diameter (D_{90}) due to the smoothing of the dataset by the software (Ibanez Sanz 2018). However, this technique was used in this study because it is very easy and fast to use. The organic matter content of the sediments was determined using an ISO standard 10694:1996-08 (ISO. 1995). The characteristics of the chosen mud samples are summarised in Table 1. Before the rheological experiments, all the mud samples were homogenized by mild hand stirring.

Rheological experiments were performed using a HAAKE MARS I rheometer (Thermo Scientific, Germany) with concentric cylinder (Couette) geometry (CC25DIN, gap width = 2 mm, distance from the bottom of cup = 5.3 mm, and sample volume = 16 ml). A waiting time of 3–5 min was used to eliminate the disturbance created by the bob after attaining its measurement position. The temperature was maintained at 20 °C during each experiment using a Peltier controller system. Each experiment was carried out in duplicate to check the repeatability of the measurements. Stress ramp-up tests were performed using the stress-controlled mode of the rheometer. An increasing stress was applied from 0 to 500 Pa at a rate of 1 Pa/s, depending upon the consistency of the sample. The corresponding motor dispalcement was measured, and the shear rate and viscosity were then determined. The flow curve experiments were carried out using a shear rate–controlled mode of the rheometer by linearly incrasing the shear rate from 0 to 25 s^{-1} in 170 s and from 25 to 300 s^{-1} in 100 s without giving enough time between each point of measurement to reach steady state. The steady state was not achieved in the flow curves to reduce the experimental time, in order to minimise the settling phenomenon which is very significant for fluid mud samples. Thixotropic experiments were performed by increasing the shear rate from 0 to 100 s^{-1}

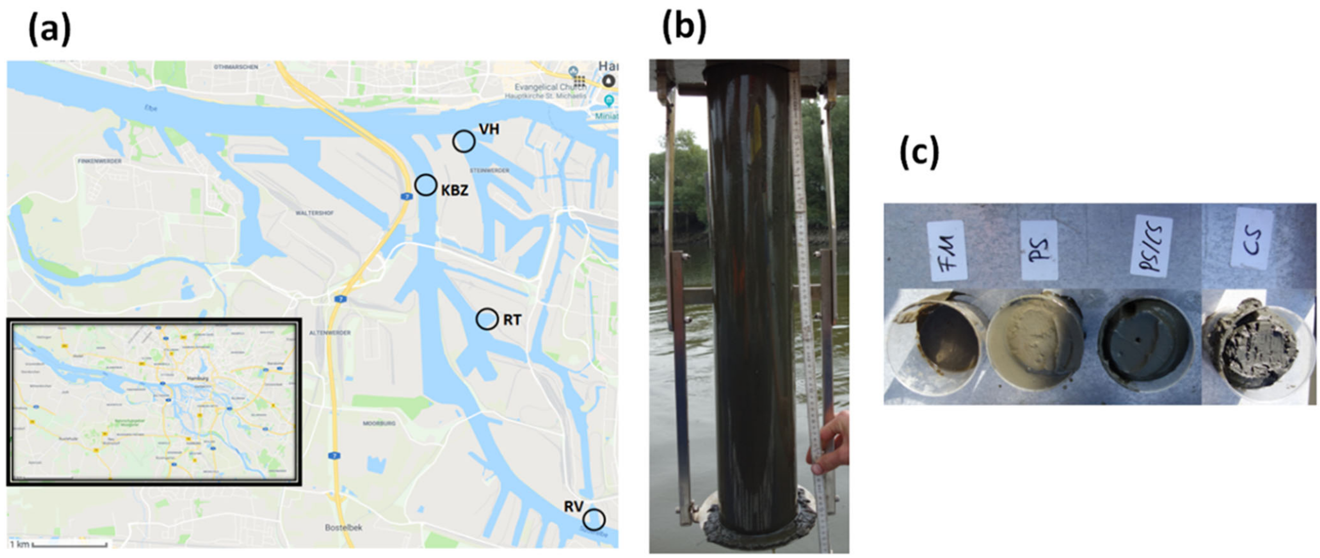

Fig. 1 **a** Selected locations in the Port of Hamburg, Germany to collect mud sample; **b** sampler of mud sediments; **c** four layers of mud samples having different visual consistencies

followed by shearing at 100 s^{-1} for 30 s and then decreasing from 100 to 0 s^{-1}. The dynamic experiments were executed either as a function of oscillation amplitude or frequency. Preliminary amplitude sweep tests were carried out at a constant frequency of 1 Hz to estimate the linear viscoelastic regimes. Frequency sweep tests were then performed from 0.1 to 100 Hz within the linear viscoelastic regimes. The storage modulus (G'), and loss modulus (G'') were recorded as a function of frequency. The complex modulus, G^* and the phase angle, δ can then be calculated as follows:

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

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

Results and discussion

Figure 2a and b presents the results of stress ramp-up tests for sediments having similar densities in the form of apparent viscosity as a function of stress. From these viscosity curves, two yield regions were identified from the decline in viscosity. However, 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 the first decline are referred to as "static" yield stress, τ_y^s, while the second decline stress values are termed as "fluidic" yield stress, τ_y^f (Shakeel et al. 2019a). The static yield stress value is most probably associated with the relaxation of the sample structure, including the breakage of large flocs into smaller flocs, as already suggested by Nosrati et al. 2011. The further breakdown of smaller flocs into very small flocs or individual particles can be linked with the fluidic yield stress values. Table 1 shows the results of stress ramp-up tests, where the mud samples with similar densities display markedly different static and fluidic yield stress values.

Italicized entries show the samples with similar higher densities and the bold entries represent the samples with similar lower densities

The samples from RV and RT revealed higher yield stress values, both static (9 Pa and 60 Pa) and fluidic (40 Pa and 312 Pa), as compared to the static (0.8 Pa and 18 Pa) and fluidic (2.44 Pa and 79 Pa) yield stress values of samples from

Table 1 Characteristics of the investigated mud samples

Sample ID	Bulk density (kg/m^3)	D_{50} (μm)	TOC (% TS)	Static yield stress (Pa)	Fluidic yield stress (Pa)	Fluidic/static yield stress (−)	Complex modulus @ 1 Hz (Pa)	Phase angle @ 1 Hz (°)
VH_FM	**1087**	**18.5**	**3.7**	**0.8**	**2.44**	**3.0**	**13 ± 0.2**	**11 ± 0.5**
RV_PS	**1098**	**25.4**	**7.2**	**9**	**40**	**4.4**	**417 ± 0.3**	**8 ± 0.5**
KBZ_PS	*1211*	*16.9*	*2.8*	*18*	*79*	*4.4*	*1774 ± 102*	*8 ± 0.7*
RT_CS	*1210*	*17.1*	*4.3*	*60*	*312*	*5.2*	*7909 ± 137*	*8 ± 0.8*

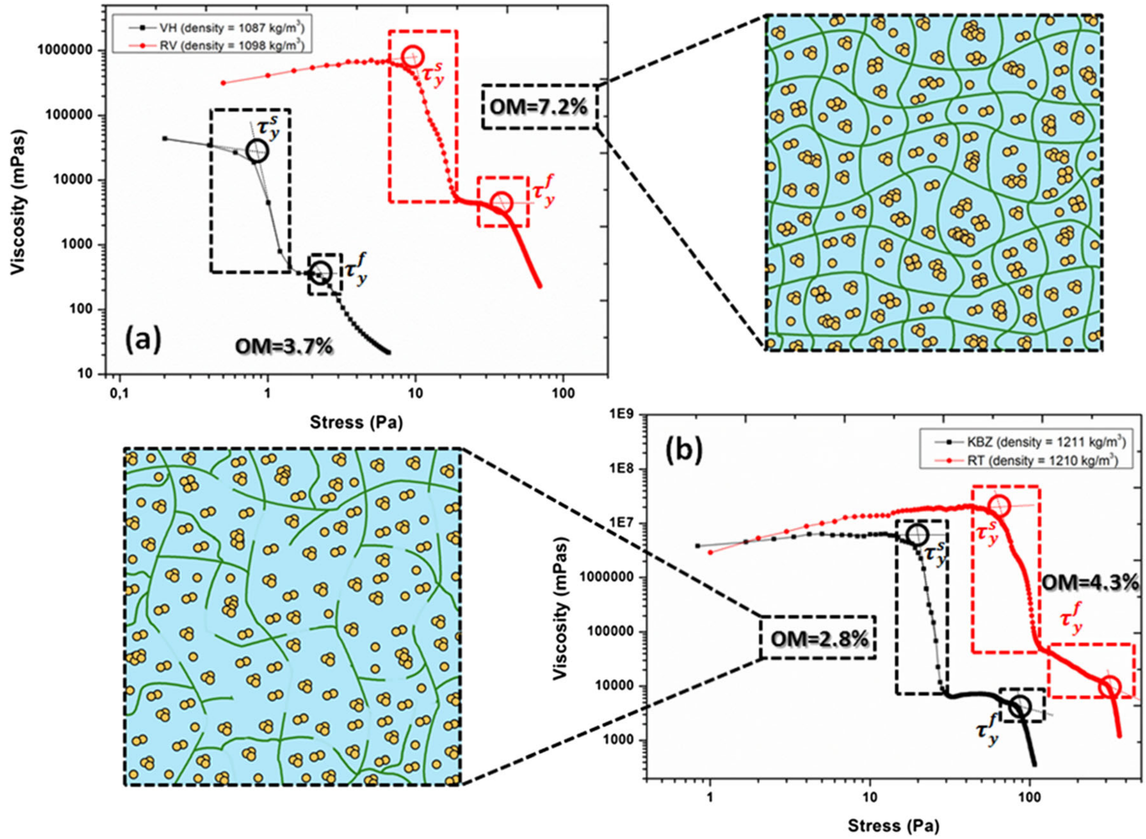

Fig. 2 Apparent viscosity as a function of stress for mud sediments having **a** similar lower and **b** similar higher densities. The boxes with dotted lines represent the yield regions and the circles represent the static (τ_y^s) and fluidic (τ_y^f) yield stress values obtained by linear slope extrapolation

locations VH and KBZ. This shows that, for lower density samples, the ratio of fluidic to static yield stress increased from 3 to 4.4 while it increased from 4.4 to 5.2 in case of higher density samples, by increasing the organic matter content. Higher yield stresses, at a given density, can be linked with higher organic matter content as a large number of flocs are strongly interconnected at high organic matter content (see Fig. 2). Increasing shear rate sweeps were also performed to obtain the flow curves. Each displayed measuring point was recorded 0.4-7 sec after the corresponding shear stress was applied. This time was too short to enable the system to reach steady state. The resultant flow curves are shown in Fig. 3a, b. The behaviour of the samples from RV and RT locations was again quite different from their corresponding location with same density, with higher stress values in response to the applied shear rate, which may be linked with their higher organic matter content.

Thixotropic behaviour of mud samples was systematically studied by increasing and decreasing the shear rate between 0 and 100 s^{-1}. The shape of the hysteresis loop depends upon various factors including the nature of the material, shear history prior to the analysis, level and rate of shearing. Comprehensive reviews have been made on thixotropy by (Barnes 1997) and (Mewis and Wagner 2009). Figure 4a, b shows the results of thixotropic experiments for the sediments having similar densities. Mud samples from RV and RT locations displayed a typical thixotropic behaviour (decrease in viscosity with shearing action), whereas anti-thixotropy or negative thixotropy behaviour was evident at lower shear rates for the sediments collected from VH and KBZ locations.

The similar combination of thixotropy and anti-thixotropy was also reported by (Nosrati et al. 2011) for muscovite dispersions. This peculiar combination of thixotropy and anti-thixotropy could be linked with the existence of shear thickening phenomenon, which was observed for these samples in stress weep tests at higher stresses. However, it could also be due to the slippage between the bob and the sample. The mud samples from RV location showed a typical thixotropic behaviour for the entire investigated shear rate range by having lower viscosity/stresses in the ramp down curve. This behaviour may again be associated with their high organic content: a large number of flocs have been disrupted which require long times to reform. These results confirm that the thixotropic behaviour of natural sediments is a strong function of organic matter content.

In the oscillatory mode of analysis, amplitude sweep tests at a constant frequency were performed prior to the frequency sweep experiments in order to identify the linear viscoelastic

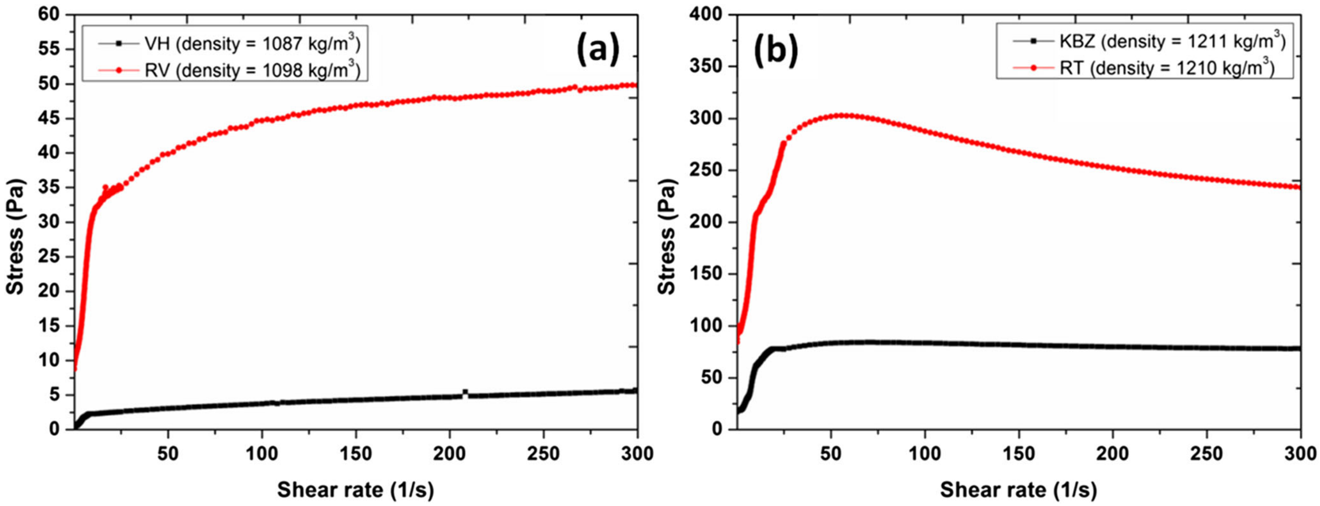

Fig. 3 Flow curves for mud sediments having **a** similar lower and **b** similar higher densities

regime for selecting suitable stress values. The results of frequency sweep tests within linear regime are shown in Fig. 5a, b, c, d. These tests are suitable for analysing the mechanical properties of the material without affecting the structure of the system. It was observed that the complex modulus of all the samples displayed a very weak frequency dependency. Furthermore, the phase angle values were very small (no cross-over), which confirmed the solid-like behaviour of the samples over the entire range of investigated frequencies. A similar solid-like behaviour of the natural sediments as a

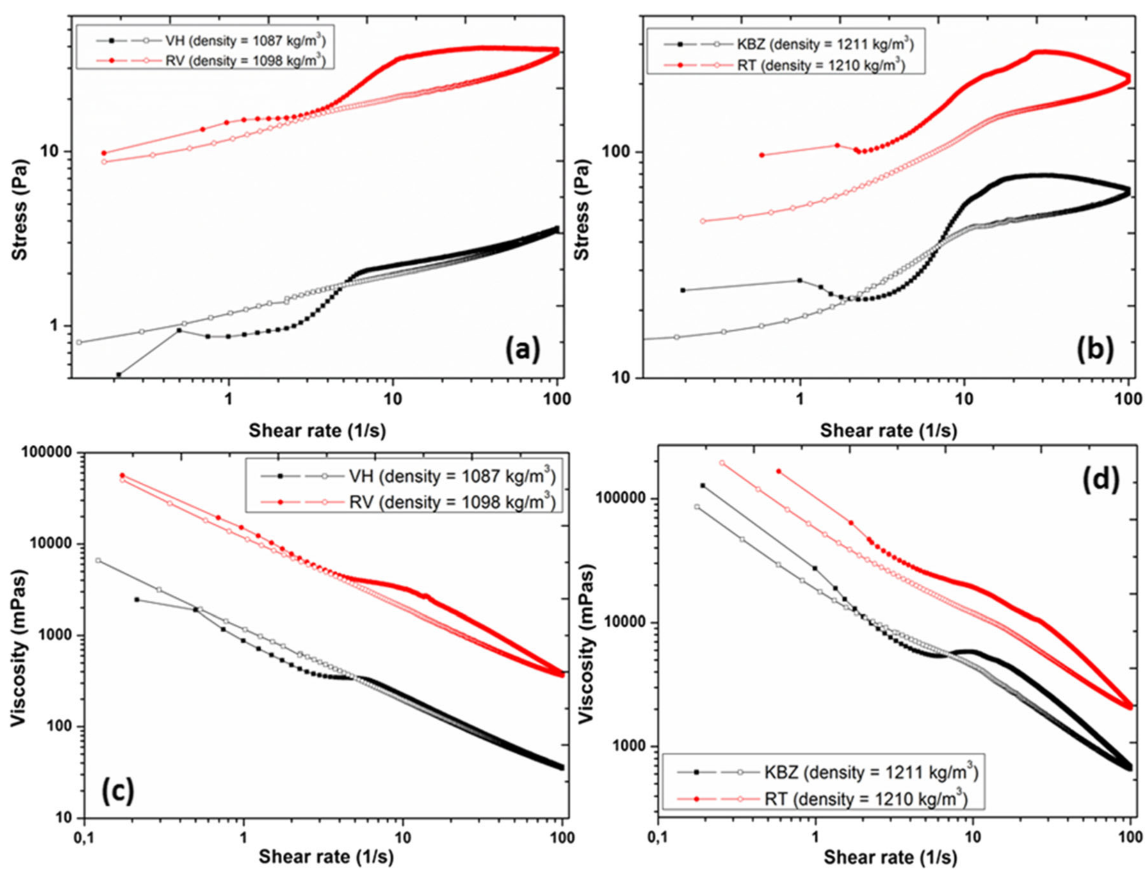

Fig. 4 **a, b** Stress as a function of shear rate and **c**, **d** apparent viscosity as a function of shear rate for mud sediments having **a, c** similar lower and **b**, **d** similar higher densities. Filled symbols represent the shear rate ramp-up and the empty symbols represent the shear rate ramp-down experiment

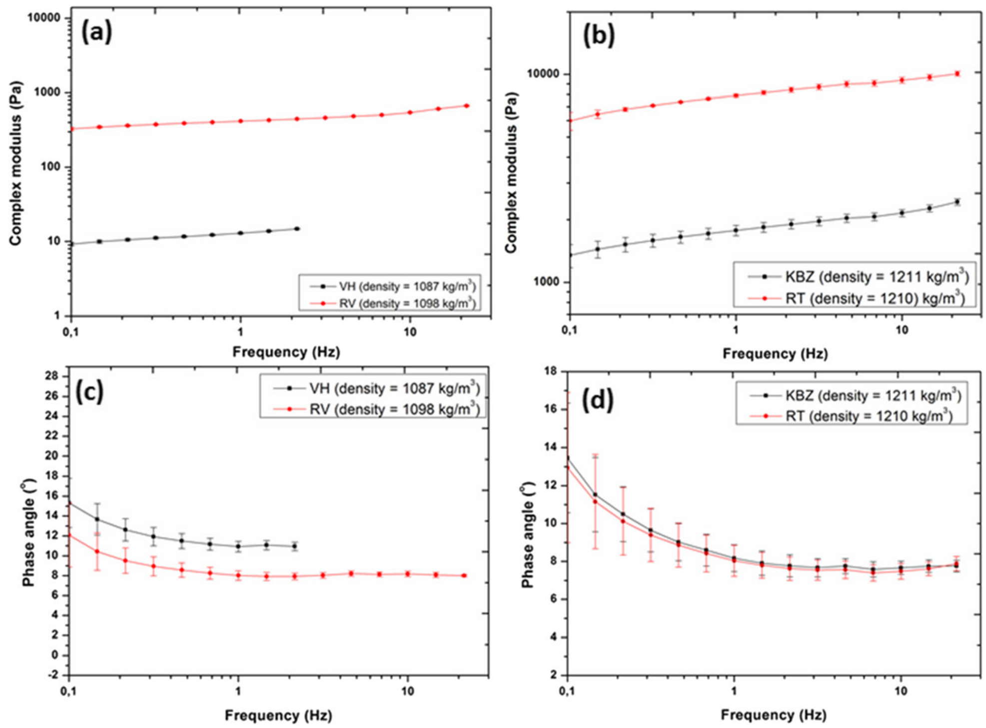

Fig. 5 **a, b** complex moduli and **c, d** phase angle as a function of frequency for mud sediments having **a, c** similar lower and **b, d** similar higher densities. Bars represent standard deviation. Solid line is a guide for the eye

function of frequency, within the linear regime, was previously reported in the literature (Van Kessel and Blom 1998; Soltanpour and Samsami 2011; Xu and Huhe 2016).

The complex modulus values were considerably different for the mud samples with similar densities, whereas the phase angle values were not markedly different from each other (see Table 1). Mud samples having higher organic matter content (RV and RT) had a higher complex modulus (417 Pa and 7909 Pa) than the ones with lower organic matter content (13 Pa and 1774 Pa), for a given density. The complex modulus is a measure of the consistency of the system, while the phase angle is representative of the degree of structuration (Lupi et al. 2016; Shakeel et al. 2018; Shakeel et al. 2019b; Shakeel et al. 2019c). The results showed that the consistency of the mud samples can be varied by changing the OM content or density, whereas the degree of structuration remains the same for all the samples. This also means that the density of all investigated samples is higher than the one required to form a structured system. At high frequencies, an increase in complex modulus and a decrease in phase angle was observed,

Table 2 Comparison of rheological properties of mud samples from different studies with this study

Study area	Density range (kg/m^3)	Fluidic/Bingham yield stress range (Pa)	Storage modulus range @ 1 Hz (Pa)	Ref.
Port of Rotterdam, the Netherlands	1168*	7	45	(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	0.02–15	(Huang and Aode 2009)
Mouth of Yangtze River, China	1650–1700	910-2810	–	(Yang et al. 2014)
Shoal of Hangzhou Bay, China	1705–1741	772–2140	–	(Yang et al. 2014)
Yangcheng Lake, China	1651–1691	2070–3960	–	(Yang et al. 2014)
Lianyungang, China	1098-1305	0.098–28.029	2–1050	(Xu and Huhe 2016)
Port of Santos, Brazil	1085–1206	5–334	–	(Fonseca et al. 2019)
Port of Rio Grande, Brazil	1132–1308	5–350	–	(Fonseca et al. 2019)
Port of Itajaí, Brazil	1138–1360	5–299	–	(Fonseca et al. 2019)
Amazon South Channel	1293–1512	5–379		(Fonseca et al. 2019)
Port of Hamburg, Germany	1087–1210	2.44-312	0.47-7915	This study

*Calculated from mud concentration

which was due to the rheometer head inertial effects at such higher frequencies. Additionally, this head inertial effect was less evident for the sample with higher organic matter content (RV) because this sample was more consistent, as shown by higher moduli (Fig. 5a). This experimental data obtained due to the head inertial effects was removed from the figures to eliminate the misconception.

The values of the rheological properties of mud samples from different locations were also compared with the rheological parameters of the mud samples investigated in this study (Table 2). The mud samples from the Port of Rotterdam, the Port of Santos and the Port of Hamburg have similar rheological parameters values for similar density ranges. However, the samples from Eckernförde Bay exhibited significantly lower yield stresses values for comparable density ranges. This might be due to the differences in organic matter content, measuring technique or analysis of rheological data. The samples from other sources displayed considerably higher rheological parameters values but their densities were much higher than the densities of the three ports mentioned above.

In literature, it is mentioned that the rheological properties particularly yield stress can be used as a criterion to define the nautical bottom (i.e. navigable fluid mud layer). For example, 100 Pa yield stress value is being used for Port of Emden, Germany as a criterion for the nautical bottom approach (Wurpts 2005). However, as the results of this study clearly indicate, for the Port of Hamburg, one yield stress value as criterion for nautical bottom, for the whole Port, would be misleading. As shown in the article, totally different rheological behaviours are observed in different regions of the Port of Hamburg, primarily because of their different content of organic matter. The definition of a critical yield stress value is therefore to be studied more into details, for different mud sample compositions to be found in a given port.

Conclusions

The mud samples analysed in the article exhibit complex rheological behaviours like viscoelasticity, shear-thinning, (anti-)thixotropy and yield stress. These rheological behaviours, and in particular, the two yield stresses found in the mud systems, are strong functions of density or volume fraction of solids. It was shown that even a few percent of organic matter affects significantly the rheological fingerprint of the mud. Mud samples having similar densities were significantly rheologically dissimilar from each other due to the presence of organic matter. The ratio of fluidic to static yield stress displayed an increase from 3 to 4.4 for samples with the density of about 1090 kg/m^3, while for higher density samples (about 1210 kg/m^3) it increased from 4.4 to 5.2, by increasing the organic matter content. From frequency sweep tests, it was shown that the complex modulus of samples with higher organic matter content (RV and RT) displayed higher values (417 Pa and 7909 Pa) than the samples with lower organic matter content (13 Pa and 1774 Pa), for a given density. This is coherent with the viscoelastic properties of organic matter/polysaccharides, already reported in literature (Baravian et al. 2007), which clearly plays a dominant role in clay-organic matter systems, even at low organic matter content. The presented analysis shows that the rheological properties of mud samples are strongly correlated with the density and organic matter of the samples. Furthermore, even small amounts of organic matter content change significantly the mud rheological behaviour. A further quantification of the effect of the type of organic matter/polyelectrolyte at different ionic strengths or pH on the rheological fingerprint of mud samples can help to link rheology to the mud density and organic matter.

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