Rheology and yielding transitions in mixed kaolinite/bentonite suspensions

Shakeel, A.; Kirichek, Alex ; Chassagne, C.

Important note
To cite this publication, please use the final published version (if applicable).
Please check the document version above.

Rheology and yielding transitions in mixed kaolinite/bentonite suspensions

Ahmad Shakeel [a,b,*], Alex Kirichek [a], Claire Chassagne [a]

[a] Faculty of Civil Engineering and Geosciences, Department of Hydraulic Engineering, Delft University of Technology, Stevinweg 1, 2628, CN, Delft, the Netherlands
[b] Department of Chemical, Polymer & Composite Materials Engineering, University of Engineering & Technology, New Campus, Lahore 54890, Pakistan

ABSTRACT

Kaolinite suspensions are known to have quite fast settling behaviour and Newtonian character, particularly at low concentrations, due to the non-swelling nature of their constitutive particles. On the other hand, bentonite is known for its swelling and interacting nature, which leads to the formation of a network structure, even at low concentrations. Therefore, both these clays are known to have significantly different rheological fingerprint. In this study, the yielding transitions in mixed kaolinite/bentonite suspensions have been investigated by varying the kaolinite/bentonite ratio and the total solid content. The detailed rheological analysis of these suspensions was carried out using amplitude sweep tests, frequency sweep tests, stress ramp-up tests and structural recovery tests.

The results showed that the kaolinite suspensions exhibited a two-step yielding behaviour in stress and amplitude sweep tests at higher solid content ($\geq$ 35 wt%). On the other hand, the bentonite suspensions displayed a single-step yielding even at higher concentrations. In mixed kaolinite/bentonite suspensions, a clear transition between a single and two-step yielding behaviour was observed in steady and oscillatory tests as a function of kaolinite/bentonite ratio, for a particular total solid content. The structural recovery experiments showed that for mixed kaolinite/bentonite suspensions at lower total solid content, where two-step yielding was present, the structural recovery was lowest. Furthermore, these results showed that the rheological properties (i. e., moduli) of stable kaolinite/bentonite suspensions were completely recovered to their initial state, even after multiple shearing cycles. The settling column analysis displayed that a very small amount of bentonite was needed to stabilize the kaolinite suspensions, which is necessary for preparing stable clay based formulations.

1. Introduction

Kaolinite and bentonite clays are widely used for different commercial applications including drilling fluids, slurry walls, cosmetics, adhesives, pharmaceuticals, refractories, etc. (Prasad et al., 1991). Both these clays are also found in quite large amount in oil-sand tailings and mine tailings, which poses serious environmental problems as their presence hamper the dewatering of these tailings (Chalaturnyk et al., 2002). In addition, both kaolinite and bentonite clays constitute a major part of the natural mud present in ports and waterways (Coussot, 1997; Shakeel et al., 2019a, 2020b, 2020c). In terms of rheology, the clays exhibit a significantly different rheological fingerprint due to the differences in particle charges, morphology of the clay platelets, presence of impurities, etc. (Au and Leong, 2013).

Bentonite suspensions display thixotropic behaviour along with high values of viscosity and yield stresses, even at low volume fraction of solids (Abend and Lagaly, 2000; Goh et al., 2011), due to the swelling nature of Na-montmorillonite present in bentonite (Lagaly, 1989). Water molecules penetrate into the interlayers of clay platelets by osmosis and cause the hydration of the sodium ions present, which results in the swelling and delamination of clay platelets. This delamination leads to an increase in particle concentration, a decrease in clay platelet size and an increase in particle's specific surface area. The high particle concentration associated with a high surface area then usually results in an enhanced particle-particle interactions which finally produces a suspension with high yield stress values (Laird, 2006; Leong et al., 1995). In contrast, kaolinite (or kaolin) suspensions display a non-Newtonian behaviour (as they have a yield stress) at high solid content (around 30–40 wt%). This behaviour is attributed to the non-swelling nature of kaolinite, as the particles do not delaminate and, hence, their surface area remains lower in comparison to montmorillonite particles, leading to limited particle-particle interactions (Teh et al., 2009). This striking difference between the two clays can be exploited by mixing them in specific ratios, in order to tune the rheological

properties of the mixture for a given particular application. For instance, the incorporation of bentonite into kaolinite suspensions has already been used to tailor the rheology of resultant system for Hanford River Protection Program (Daniel and Eibling, 2005).

Rheology is one of the most important techniques to determine the interactions between the particles in a suspension and even a small change in the composition of the system can lead to a significantly different rheological fingerprint (İşci et al., 2004; Shakeel et al., 2019b). At low shear, the viscosity of a suspension is primarily controlled, besides particle concentration, by colloidal properties such as surface charge, particle size distribution and suspending medium properties such as pH and salinity. However, at high shearing conditions, the viscosity is determined by the particle size and its distribution, particle shape and the arrangement of particles in the suspension (Czibulya et al., 2010). The rheological behaviour of kaolinite and bentonite suspensions has been extensively studied in literature as a function of several factors including pH (Kelessidis et al., 2007; Palomino and Santamarina, 2005), source of clay (Melton and Rand, 1977; Teh et al., 2009), ionic concentration (Johnson et al., 1999), type of ions (Montes-H et al., 2003; Mpofu et al., 2003), additives and flocculants (Leong et al., 2012; Mpofu et al., 2004; Shakeel et al., 2020e; Yalçin et al., 2002). However, the rheological fingerprint of mixed kaolinite/bentonite suspensions has not been investigated in detail (Adebayo and Ajayi, 2011; Au and Leong, 2013; Grabowska-Olszewska, 2003; Kasperski et al., 1986; Keren, 1989; Lagaly, 1989) and, hence, a complete understanding of these mixed suspensions is lacking.

It has been reported in literature that the incorporation of small amount of bentonite in kaolinite suspensions can create a significant change in the rheological properties of the resultant suspension. For instance, mixed kaolinite/bentonite suspension can display a similar rheological fingerprint to kaolinite suspensions, but at a much lower total solid content (Keren, 1989; Lagaly, 1989). Moreover, a Newtonian behaviour of kaolinite suspensions at low solid content (5 wt%) can be transformed into a non-Newtonian behaviour by adding a small amount of bentonite/montmorillonite (Keren, 1989). This change in rheological properties of suspensions, by adding bentonite, becomes more prominent by reducing the pH from 7 to 4, which is attributed to the increased interactions between the clay particles (Keren, 1989). On the contrary, addition of kaolinite into bentonite suspensions resulted in lower viscosity for higher density of the resultant system, which is quite important for drilling fluid formulations (Adebayo and Ajayi, 2011). Au and Leong (2013) showed that the magnitude of yield stress was determined by the bentonite content while the location of maximum point of yield stress vs pH curve was controlled by the kaolinite amount for mixed kaolinite/bentonite suspensions (Au and Leong, 2013). However, the yielding transitions (i.e., single-step or two-step) in mixed kaolinite/bentonite suspensions have never been studied in literature, according to our knowledge. Furthermore, the corresponding study of the settling behaviour of these mixed suspensions is still missing. The main objective of the present article is, therefore, to investigate the yielding transitions in mixed kaolinite/bentonite suspensions, as a function of total solid content and the kaolinite/bentonite ratios. The rheological analysis of individual clay suspensions was also performed, as the source of clay can significantly affect the rheology of the suspension. A detailed rheological analysis of individual and mixed clay suspensions was carried out with the help of amplitude sweep tests, frequency sweep tests, stress ramp-up tests and structural recovery tests. The effect of bentonite addition on the settling behaviour of kaolinite suspensions was investigated and discussed in correlation with the rheological studies.

2. Experimental

2.1. Synthesis of suspensions

Kaolinite (provided by Imerys, England) and Na-bentonite (also known as Cebogel OCMA having 70% of montmorillonite, supplied by

Table 1
Mineralogical composition of bentonite (taken from Kiviranta and Kumpulainen, 2011).

Mineral	wt%
Clay minerals	*72.7*
1. Smectite	69.1
2. Illite	3.6
3. Biotite/chlorite	Traces
Feldspars	*4.0*
Tridymite/opal	*1.0*
Quartz	*0.6*
Iron oxides	*0.5*
1. Hematite	0.2
2. Geothite	0.3
Pyrite	*1.3*
Anatase	*0.3*
Soda (hydrated)	*2.3*
Carbonates	*17.4*
1. Calcite	13.4
2. Dolomite	4.0

Cebo Holland BV, the Netherlands) based suspensions were prepared by dispersing different amounts of clays (see Tables S1 and S2, which can be found online in Supplementary Material) in deionized water (Merck Millipore, Darmstadt, Germany). The mineralogical and chemical composition of the bentonite is taken from the literature (Kiviranta and Kumpulainen, 2011), as they also used the same bentonite (see Table 1 and Table S3). The specific weight and specific surface of the considered bentonite is 2837 kg. m^{-3} and 130 m^2. kg^{-1}, respectively (Kiviranta and Kumpulainen, 2011). The cation exchange capacity of used bentonite is 0.89 eq/kg with the following exchangeable cations composition: Ca^{+2} = 0.05, K^+ = 0.02, Mg^{+2} = 0.06 and Na^+ = 0.90 (Kiviranta and Kumpulainen, 2011). On the other hand, the specific weight and specific surface of the kaolinite is 2695 kg. m^{-3} and 18 m^2. kg^{-1}, respectively. In order to obtain a homogenous system, a high shear mixing was performed at 1000 rpm for 5–10 min using an IKA homogenizer (RE 162, IKA-Werke GmbH & Co., Staufen, Germany). All the suspensions were left for 24 h at 20 °C, in order to achieve the complete swelling of montmorillonite present in bentonite. The pH of the kaolinite suspension was found to be within the range of 5–6. The pH of the bentonite suspension was observed to be around 8, while in case of mixed kaolinite/bentonite suspension the pH value was about 7–7.5.

2.2. Particle size distribution (PSD)

The particle size distribution of the prepared suspensions was analysed by using static light scattering technique (Malvern MasterSizer 2000MU). This instrument is based on the Mie theory which basically assumes the spherical shape of all the particles (Ibanez Sanz, 2018), which can be inappropriate for the considered clays. However, this technique was used in this study because it is very easy and fast to use and gives reasonable idea about the particle size and its distribution. The results for kaolinite suspensions are presented in Fig. S1a and Table S4. The median particle size (D_{50}) of the bulk kaolinite suspension (used for further experiments) was observed to be 8.14 ± 0.03 μm. However, a small peak was clearly evident around 1 μm which could be due to the non-spherical shape of the particles. Therefore, the particle size distribution of supernatant liquid was also performed after the settling of larger kaolinite particles (see Fig. S2a). In the supernatant case, the D_{50} was around 3.4 μm, which confirmed the existence of smaller kaolinite particles within the sample, in addition to their non-sphericity.

In order to analyse the effect of particle size distribution on the rheological fingerprint of bentonite suspensions, dry bentonite was sieved using a sieve of 53 μm mesh size. The particle size distributions of bentonite without sieving (stated as 'M'), smaller than 53 μm (stated as 'S') and larger than 53 μm (stated as 'L') are presented in Fig. S1b and Table S4. It can clearly be seen from the Fig. S1b that bentonite particles
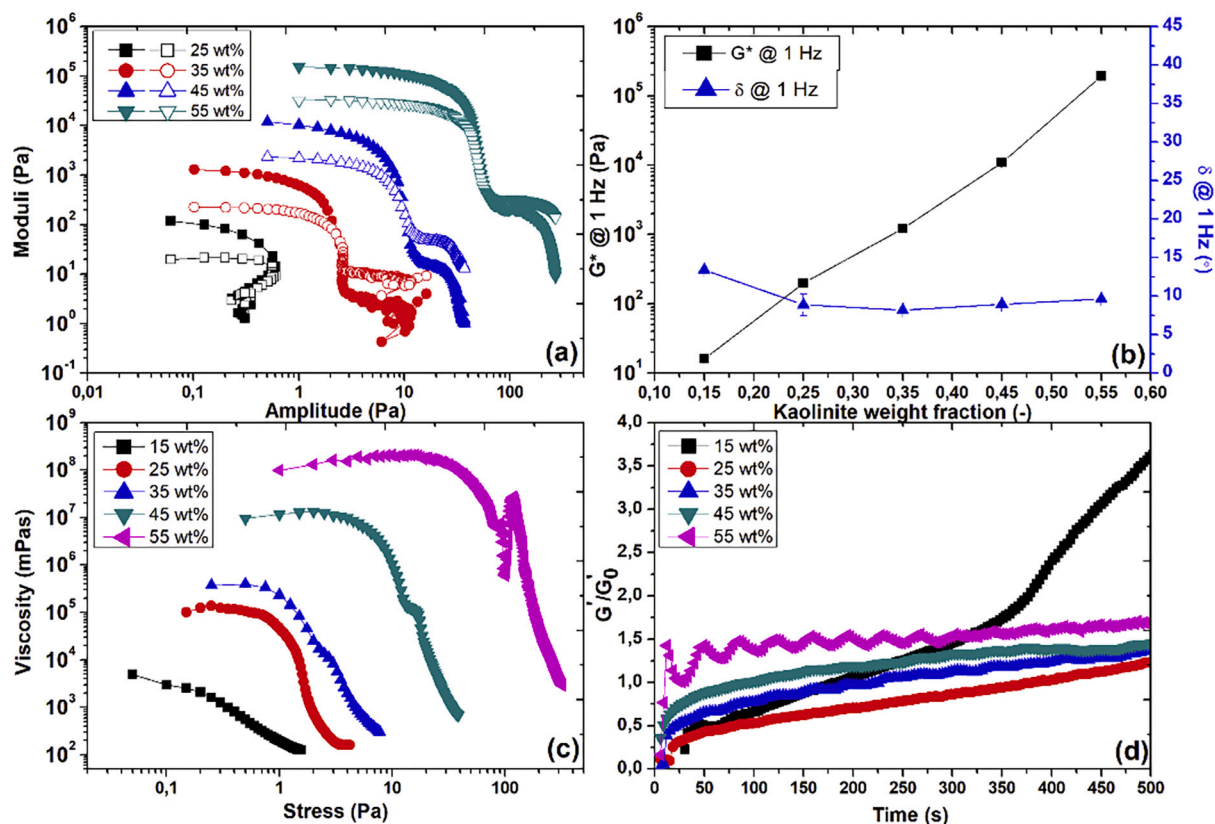
Fig. 1. (a) Storage modulus (filled symbols) and loss modulus (empty symbols) as a function of amplitude for suspensions with different amount of kaolinite obtained at 1 Hz, (b) complex modulus and phase angle at 1 Hz as a function of kaolinite fraction, (c) apparent viscosity as a function of stress for suspensions with varying kaolinite amount, and (d) normalized storage modulus (G'/G_0') as a function of time for suspensions with varying kaolinite amount obtained from the structural recovery step. Solid line is just a guide for the eye. Bars represent standard deviation.

smaller than 53 μm displayed peaks at large particle sizes, which may be due to the polydispersity of particles, anisotropic shape of particles or presence of flocs. This peak at large particle size may also be attributed to the smoothing of the dataset by the Malvern software (Ibanez Sanz, 2018). The particle size distribution of some mixed kaolinite/bentonite suspensions was also analysed and the results are displayed in Fig. S2b and Table S4. The results showed an increase in the D_{50} value of the suspensions by increasing the bentonite content, which can be linked to the larger bentonite particles.

2.3. Rheological analysis

The rheological characteristics of individual and mixed kaolinite/bentonite suspensions were determined by using a HAAKE MARS I rheometer (Thermo Scientific, Germany). Vane-in-cup geometry, with a gap width of 2.5 mm, was used to perform rheological experiments, in order to prevent slippage problem (Barnes, 1995). The repeatability error was estimated by performing all the experiments in duplicate and it was always less than 2%. Peltier controller system was used to regulate the temperature during all the experiments. First of all, oscillatory amplitude sweep experiments were performed to investigate the moduli behaviour (i.e., solid-liquid transition represented by the crossover between G' and G'') as a function of applied amplitude and also to determine the linear viscoelastic (LVE) regime of prepared suspensions, required for performing the frequency sweep tests. The amplitude sweep tests were carried out at a constant frequency of 1 Hz for individual and mixed kaolinite/bentonite suspensions. All the experiments were carried out at a constant temperature of 20 °C.

Frequency sweep tests were performed for the prepared suspensions, within LVE regime, to analyse the strength of the suspension without

affecting its structure. The frequency was varied from 0.1 to 100 Hz and the results were presented in the form of complex modulus (G^*) and phase angle (δ) as a function of frequency. The yield stress of the suspensions was determined by performing the stress ramp-up tests, i.e., linear increase in stress at a rate of 0.5 or 1 Pa/s, until the shear rate reaches 100 s^{-1}. The torque was recorded as a function of applied stress, which was then represented in the form of apparent viscosity. The structural recovery test was performed for the prepared suspensions by following the protocol reported in (Shakeel et al., 2020a). The first step involved the application of an oscillatory time sweep test within LVE regime for 100 s, to estimate the moduli of suspensions before structural breakup. A pre-shearing step was then performed at 100 s^{-1} for 500 s, followed by a non-destructive recovery step at 1 Hz for 500 s (see Fig. S3). Although for bentonite suspensions the recovery process can last for long time, the recovery step was performed for 500 s which was enough to understand the recovery behaviour of suspensions as a function of different parameters. The storage modulus $G'(t)$ was recorded as a function of time in the recovery step. Moreover, a stretched exponential functional was used to quantify the structural recovery parameters, given as follows (Mobuchon et al., 2007, 2009):

$$\frac{G'(t)}{G_0'} = \frac{G_i'}{G_0'} + \left(\left(\frac{G_\infty' - G_i'}{G_0'} \right) \left(1 - exp\left[-\left(\frac{t}{t_r} \right)^d \right] \right) \right) \quad (1)$$

where $G'(t)$ is the time dependent storage modulus of suspensions in the recovery step, G_0' is the initial storage modulus before shearing, G_i' is the storage modulus directly after the shearing step, G_∞' is the equilibrium storage modulus, t_r represents the characteristic time of the suspensions and d is the stretching exponent. G_∞', t_r and d are the fitting parameters in Eq. (1).

2.4. Settling column experiments

In order to analyse the influence of bentonite addition on the stability of kaolinite suspensions, settling column experiments were performed in 500 mL glass columns with a diameter of 4.5 cm and length of about 27 cm. The experiments were conducted for different concentrations of total solids (i.e., 5, 10, 15 and 20 wt%) along with varying content of bentonite. After homogenizing the samples, the columns were filled with the suspensions and the images were taken after every 25 s during the settling phase for 24 or 48 h. The clay/water interface height was then recorded and plotted as a function of time.

3. Results and discussion

3.1. Kaolinite suspensions

3.1.1. Effect of solid content

Before analysing the characteristics of mixed kaolinite/bentonite suspensions, the rheological properties of kaolinite and bentonite suspensions were investigated. The non-Newtonian rheological behaviour of kaolinite suspensions is strongly dependent on the clay content. Therefore, a detailed rheological analysis was performed by varying the solid content of kaolinite suspensions. Fig. 1a depicts the outcome of amplitude sweep tests in terms of storage and loss moduli for kaolinite suspensions with varying solid content. A clear transition from a single-step yielding (25 wt%) to a two-step yielding (> 25 wt%) was found. This may be attributed to the fact that at low kaolinite concentrations, only diffusive dynamics were playing a role while at higher solid content both trapped and diffusive dynamics were responsible for observed rheological behaviour (Dagois-Bohy et al., 2017; Zhao et al., 2014). A similar transition between single and two-step yielding behaviour was observed for cocoa/vegetable oil suspensions as a function of solid content (Ahuja and Gamonpilas, 2017). The oscillatory strain sweep was also performed for the kaolinite suspension having 55 wt% and the result is presented in Fig. S4. It was clearly evident that the strain sweep also showed the existence of two-step yielding. The applied and measured waveforms, during these tests, were sinusoidal after the first decline. However, the waveforms were distorted after the second decline, due to high deformations.

The results of frequency sweep tests in terms of complex modulus and phase angle are shown in Fig. S5a and S5b for different kaolinite suspensions. For all the kaolinite suspensions, a solid-like behaviour was found (with almost frequency independent moduli and low phase angle values). In order to clearly quantify the effect of solid content on the modulus and phase angle behaviour, both parameter values at 1 Hz are plotted as a function of solid content (Fig. 1b). It can be clearly seen that the complex modulus showed a significant increase as a function of solid content while the phase angle decreased initially and remained more or less constant after 25 wt% of kaolinite. This behaviour showed that the consistency/strength of the system (i.e., modulus) can be tuned as a function of solid content without much affecting the degree of structuration (i.e., phase angle). The increase in moduli as a function of increasing kaolinite fraction was also observed in literature (Lin et al., 2015).

Apart from oscillatory measurements, stress ramp-up tests were also performed for kaolinite suspensions with varying kaolinite fraction. The results are presented in Fig. 1c in terms of apparent viscosity as a function of stress. These results confirmed the transition between single-step ($\leq$ 25 wt%) and two-step yielding (> 25 wt%), found using amplitude sweep tests. Moreover, at the highest solid content (55 wt%), a significant increase in apparent viscosity was observed after first yield point which may be linked to the existence of jamming phenomenon, in addition to the two-step yielding, at such a high concentration. This behaviour was further verified by performing the stress ramp-up experiments using grooved Couette geometry (data not shown), which showed the existence of jamming phenomenon.

Table 2
Rheological properties of bentonite samples with different particle size distribution.

Sample ID	G* @ 1 Hz (Pa)	δ @ 1 Hz (°)	G_∞'/G_0'	t_r (s)
Bentonite samples (not sieved)				
3 T-100B	7.7 ± 0.7	27.2 ± 3.1	–	–
5 T-100B	167.8 ± 2.1	5.0 ± 0.1	0.83	236.1
7 T-100B	1046.3 ± 36.2	2.84 ± 0.02	1.02	256.0
10 T-100B	6904.4 ± 53.8	2.86 ± 0.02	0.96	309.2
Bentonite samples (< 53 μm)				
3 T-100B	14.0 ± 0.7	12.5 ± 1.4	–	–
5 T-100B	286.6 ± 5.0	2.58 ± 0.03	0.79	210.6
7 T-100B	1516.6 ± 27.4	2.33 ± 0.01	0.83	207.2
10 T-100B	8832.4 ± 161.9	2.54 ± 0.02	0.79	207.7
Bentonite samples (> 53 μm)				
3 T-100B	12.7 ± 0.1	10.3 ± 0.9	–	–
5 T-100B	164.2 ± 1.6	5.84 ± 0.01	3.23	417.8
7 T-100B	933.1 ± 13.1	3.65 ± 0.03	1.33	355.5
10 T-100B	5445.8 ± 32.5	3.14 ± 0.01	1.03	263.9

Furthermore, in order to observe the structural recovery behaviour after shearing, kaolinite suspensions were subjected to a pre-shearing step followed by a recovery step within LVE regime. The initial storage modulus (G_0') was measured before the pre-shearing step by performing the time sweep experiment within LVE regime for 100 s. The time dependent storage modulus, $G'(t)$ was normalized with this initial value of the modulus, in order to investigate how much structure has been recovered as compared to the initial one. The results are presented in terms of normalized storage modulus as a function of time for different kaolinite suspensions (Fig. 1d). For the lowest kaolinite concentration (i.e., 15 wt%), a sharp increase in moduli was observed after 350 s, which may be attributed to the settling of kaolinite particles and the resulted increase in solid content at the bottom of the measuring cup. Therefore, kaolinite concentrations lower than 15 wt% were not selected for analysis in this study. A slight increase in modulus at larger recovery times was found for the 25 wt% sample, which was linked to the settling of kaolinite particles in this case as well. At the highest kaolinite content (55 wt%), an oscillating behaviour of moduli was observed which may be linked to the enhanced elastic character of the samples at such a high concentration. A similar oscillating behaviour of moduli in structural recovery tests was observed in literature for natural mud samples (Shakeel et al., 2020a). The value of normalized storage modulus was sometimes higher than 1, which may attributed to either longer resting time than 100 s used to measure the initial storage modulus or due to the improved structural rearrangements during shearing step. In order to quantify the structural recovery parameters, the experimental data of normalized moduli was fitted with an empirical equation (Eq. 1). The fitting parameters are presented in Table S5. The equilibrium structure (i.e., G_∞'/G_0') obtained after infinite recovery time was stronger for the samples particularly with low kaolinite concentrations (Table S5), which may be attributed to either efficient dispersion of kaolinite particles by the pre-shearing step or settling of kaolinite particles at lower concentrations. However, the recovery time to achieve 63% of equilibrium structure (i.e., t_r) was shortest for the sample having highest kaolinite fraction (i.e., 55 wt%). This result indicated that the larger G_∞'/G_0', observed in the case of low kaolinite content, was linked to the settling of kaolinite particles, which took longer time. This increase in G_∞'/G_0' may also be attributed to the polydispersity of the sample, which facilitated the re-organization of particles due to the presence of bigger and smaller particles.

3.2. Bentonite suspensions

3.2.1. Effect of particle size distribution

Bentonite based suspensions were characterized with the help of rheology. As already mentioned in the experimental section, bentonite
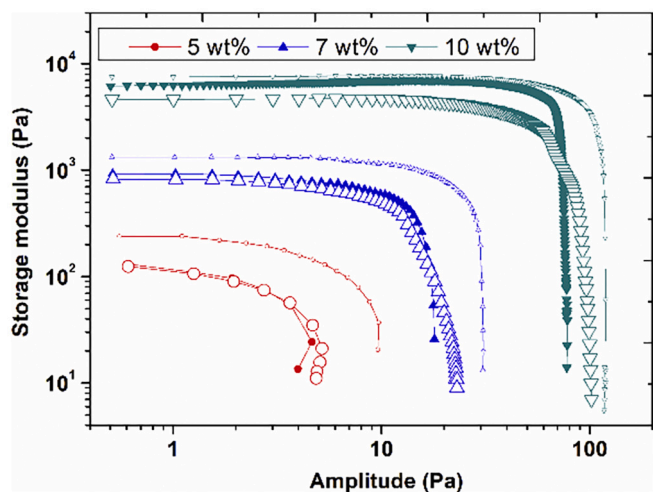
Fig. 2. Storage modulus as a function of amplitude for suspensions with different amount of bentonite obtained at 1 Hz, bigger empty symbols represent particles larger than 53 μm, smaller empty symbols represent particles smaller than 53 μm and filled symbols represent particles without sieving.

was sieved to obtain samples with different particle size distributions. The effect of particle size distribution on the rheological properties of bentonite suspensions is shown in Fig. S6. From moduli and phase angle behaviour (Fig. S6a-d, Table 2), it was clear that the suspensions having smaller particles displayed higher values of moduli and smaller values of phase angle. This stronger structure in suspensions with smaller particles may be linked to the higher surface area of the particles, which led

to enhanced particle-particle interactions and, thus, contributed to the strength of the network (Chang and Powell, 1994). A larger yield stress was also found for suspensions having smaller particles (Fig. S7a), which may be linked to the same explanation. The outcome of amplitude sweep tests for bentonite suspensions with varying particle size distribution is shown in Fig. 2. The suspensions having smaller particles showed a larger linear viscoelastic regime (LVE) (implying that the modulus remained constant as a function of amplitude). At high solid content (10 wt%), suspensions having larger particles showed a small bend (i.e., a weak two-step yielding) in their modulus curve as a function of amplitude. This peculiar behaviour may be attributed to the rearrangement of larger particles during the shearing action, which was already observed in literature for binary colloidal glasses (Sentjabrskaja et al., 2013).

The equilibrium structure obtained after the structural recovery test is presented in Fig. S7b for bentonite suspensions with varying particle sizes. It was found that suspensions with larger particles showed a stronger structure after pre-shearing action, particularly at low solid content (Fig. S7c, Table 2), which may again be linked to the reorganization and particle-particle interactions of larger particles during shearing, leading to an improved structural packing (Luckham and Ukeje, 1999). It was observed that the recovery time (t_r) was highest for the suspensions having the largest particles (Fig. S7d, Table 2). This behaviour may be linked to the fact that bigger particles require longer recovery time. While for suspensions having smaller particles, the recovery was quite fast and independent of particle concentration. The values of the fitting parameters found using Eq. 1 for bentonite suspensions with varying particle size distribution are presented in Table S6.

3.2.2. Effect of solid content

Apart from analysing the effect of particle size distribution, the effect
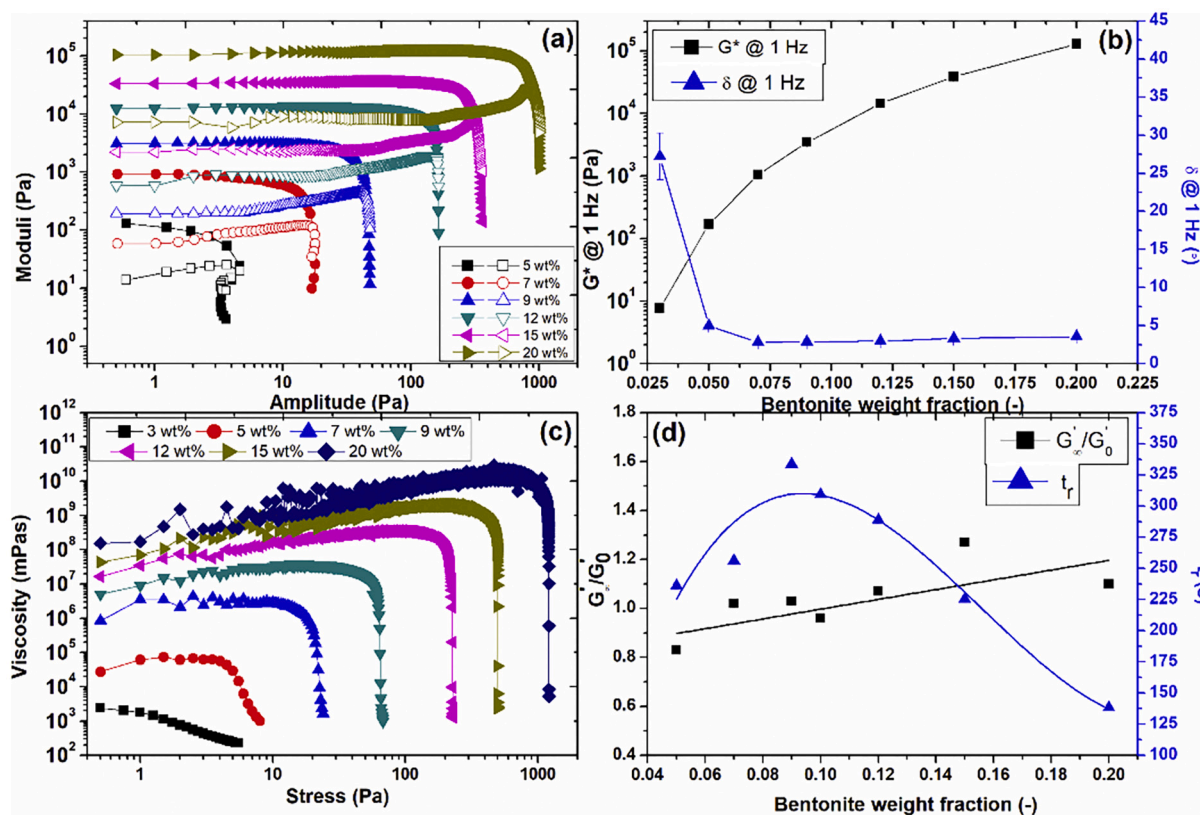

Fig. 3. (a) Storage modulus (filled symbols) and loss modulus (empty symbols) as a function of amplitude for suspensions with different amount of bentonite obtained at 1 Hz, (b) complex modulus and phase angle at 1 Hz as a function of bentonite fraction, (c) apparent viscosity as a function of stress for suspensions with varying bentonite amount, (d) equilibrium structure (G_∞'/G_0') and recovery time (t_r) as a function of bentonite content. Solid line is just a guide for the eye. Bars represent standard deviation.

of solid content on the rheological fingerprint of bentonite suspensions was also investigated. The outcome of different rheological tests for bentonite suspensions as a function different solid content is presented in Fig. 3. In Fig. 3a, it is shown that the bentonite suspensions exhibited a single-step yielding, in contrast to the two-step yielding found for kaolinite suspensions, for all the investigated solid content. This interesting behaviour may be associated to the strong interactions between montmorillonite particles (Abend and Lagaly, 2000), which resulted in a single-step structural breakup without any reorganization. Furthermore, as already observed for kaolinite suspensions, an increase in LVE regime (Fig. 3a), moduli (Fig. 3b), and yield stress (i.e., determined from viscosity decline) (Fig. 3c), were observed as a function of increasing solid content, as already observed in literature (Choo and Bai, 2015; Huang et al., 2016). The sample having lowest concentration of bentonite (3 wt %) showed lowest values of moduli and yield stress and highest values of phase angle, which was associated with the liquid-like nature of the sample. However, for 5 wt% bentonite, there was a significant increase in moduli and yield stress with small value of phase angle, which showed that the critical concentration of bentonite required to form a gel-like structure was around 3–5 wt%, for the investigated bentonite sample. This value corresponded well to the values found in literature (i. e., 3–5 wt%) for the bentonite suspensions (Huang et al., 2016; Keles-sidis and Maglione, 2008). Further rheological results for these suspensions can be found in supplementary information (Fig. S8).

The parameters for structural recovery (i.e., $G_\infty{}'/G_0{}'$ and t_r) for bentonite suspensions as a function of solid content, obtained from the recovery step (Fig. S9) after pre-shearing, are shown in Fig. 3d. It was quite interesting to see that the equilibrium structure after pre-shearing was almost similar as the initial one (i.e., the value of $G_\infty{}'/G_0{}'$ is fluctuating around 1) as a function of solid content, except for the sample with lowest solid content. This confirmed that the origin of structural network in bentonite suspensions was due to the interactions between the montmorillonite particles and when enough particles were present for interactions (i.e., to form gel-like material), the strength of the system recovers to almost the same value after pre-shearing. However, the recovery time (t_r) was varying as a function of solid content (Fig. 3d). For low solid content ($<$ 9 wt%), an increase in recovery time was observed as a function of increasing solid content while a decrease in recovery time as a function of increasing solid content was found for suspensions having solid content higher than 9 wt%. This threshold in change in recovery time was also found by Lee et al. (2012). The values of the fitting parameters using (Eq. 1) for bentonite suspensions with varying solid content are presented in Table S7.

3.3. Mixed kaolinite/bentonite suspensions

3.3.1. Amplitude sweep tests

After analysing the rheological properties of kaolinite and bentonite suspensions, different samples were prepared by mixing kaolinite and bentonite. Both total solid content and kaolinite/bentonite ratio were varied (see Table S2). The results of amplitude sweep tests for mixed kaolinite/bentonite suspensions as a function of different total solid content and kaolinite/bentonite ratio are presented in Fig. S10. All these amplitude tests were performed at a frequency of 1 Hz, in order to shorten the experimental time, which is particularly relevant for suspensions having high solid content. From Fig. S10a and S10b, it can be seen that at high kaolinite fractions or at high bentonite fractions, the material displayed either kaolinite or bentonite dominating behaviour while for intermediate ratios of kaolinite and bentonite, the system showed a peculiar two-step yielding behaviour. This transition from single-step yielding to two-step yielding behaviour may be attributed to the fact that at intermediate ratios both kaolinite and bentonite particles played their role. The reorganization between montmorillonite particles led to the first yield point and the reorganization of particles at second yield point was driven by the kaolinite particles.

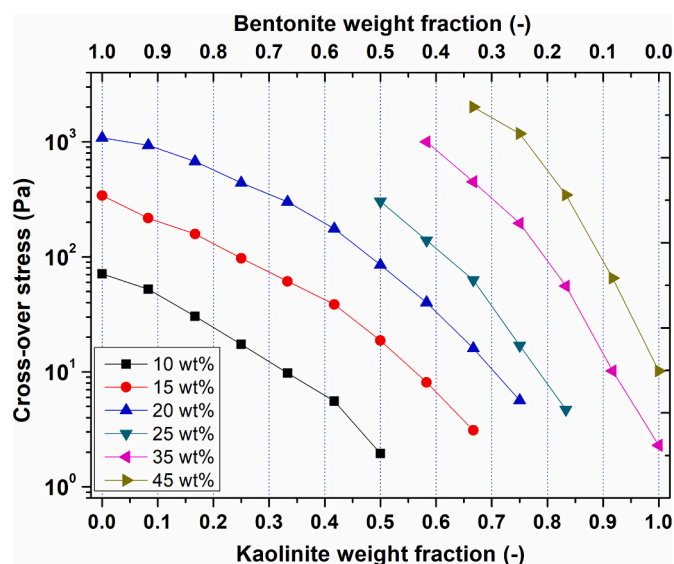This transition was not clearly visible in amplitude sweep tests for

Fig. 4. Cross-over stress as a function of kaolinite/bentonite ratio for different total solid content. Solid line is just a guide for the eye.

the suspensions having high solid content (20–35 wt%, Fig. S10c-S10e), which may be due to the fact that a high frequency of 1 Hz was used for the experiments. This resulted in inertial problems for liquid-like samples (or with weak structure) after the first yield point (Heymann et al., 2002; Läuger and Stettin, 2016). The critical stress (i.e., below which the system lies within the LVE regime) and cross-over stress (i.e., the stress at which $G' = G''$) are plotted as a function of kaolinite/bentonite fraction for different total solid content in Fig. S11 and Fig. 4, respectively. The critical stress is essential to know before performing the frequency sweep tests, as it highlights the deviation of the system from linear viscoelastic behaviour. The cross-over stress, on the other hand, is important as it represents the transition from solid-like material to liquid-like material (Werner et al., 2017). It can be seen that both parameters were significantly dependent on both total solid content and kaolinite/bentonite ratios. The samples having highest bentonite fractions for highest total solid content or samples with highest kaolinite fractions for lowest total solid content were not analysed because the samples were either too strong or too weak to analyse using this experimental method.

3.3.2. Frequency sweep tests

After analysing the amplitude sweep results for mixed suspensions, the frequency sweep tests were performed within the LVE regime, in order to investigate the strength of the network without disturbing the sample. The results of frequency sweep tests are presented in terms of complex modulus at 1 Hz (Fig. 5a) and phase angle at 1 Hz (Fig. 5b) as a function of kaolinite/bentonite fraction for different total solid content. Fig. 5a shows that the complex modulus increased as a function of increasing bentonite fraction for all the investigated solid contents. This showed that the consistency (G^*) and degree of structuration (δ) of the mixed suspension can be tuned by varying the total solid content and kaolinite/bentonite ratio. However, in the presence of small amounts of bentonite (8 wt%), the modulus decreased (Fig. 5a) and phase angle increased (Fig. 5b) compared to the value of kaolinite suspensions, for low solid content (10–25 wt%). This decrease in modulus and increase in phase angle may be attributed to the presence of slight amount of salt in bentonite (see conductivity values in Table S2), which eventually resulted in particle-particle interactions and, hence, lower moduli and higher phase angle values (Luckham and Rossi, 1999). This salt screening effect was not very significant for the samples having either high bentonite fractions or high solid content, which may be linked to the fact that (i) montmorillonite particles are known to have long-range
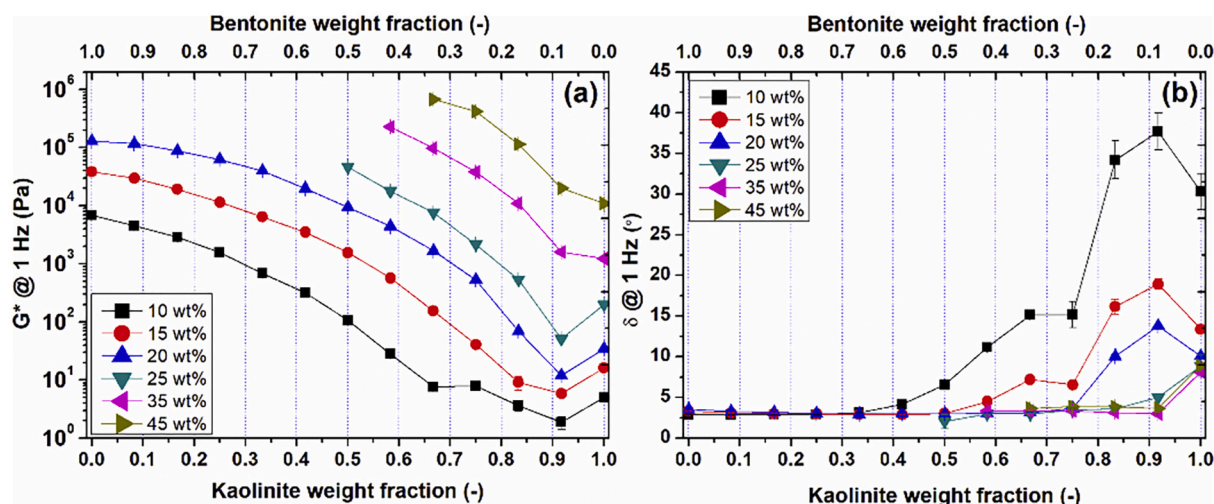
Fig. 5. (a) Complex modulus and (b) phase angle at 1 Hz as a function of kaolinite/bentonite ratio for different total solid content. Solid line is just a guide for the eye. Bars represent standard deviation.
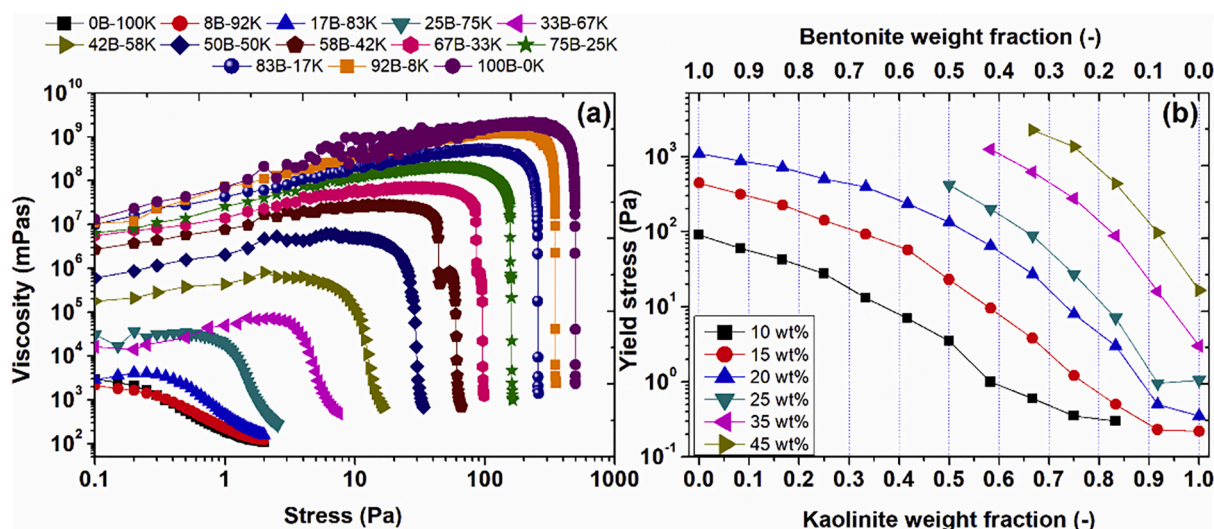

Fig. 6. (a) Apparent viscosity as a function of stress for suspensions having 15 wt% total solid content with varying ratios of kaolinite/bentonite, (b) yield stress (determined from the viscosity decline and second yield point in case of systems with two-step yielding) as a function of kaolinite/bentonite ratio for different total solid content. Solid line is just a guide for the eye.

particle-particle interactions and (ii) the interaction between montmorillonite particles increases rapidly with decreasing distance between particles (at high solid content). More rheological results for frequency sweep tests can be found in supplementary information (Fig. S12 and S13).

3.3.3. Stress ramp-up tests

Stress ramp-up tests are quite fast to perform. Yield stress values of the samples can be obtained from the viscosity declines (Shakeel et al., 2020d). Stress ramp-up tests were performed for the mixed suspensions having different total solid content and kaolinite/bentonite fractions. As for amplitude sweep tests, a clear transition from single-step yielding to two-step yielding was found at intermediate kaolinite/bentonite ratios for different total solid content, see Fig. 6a. For high kaolinite or high bentonite based suspensions, a single-step yielding was observed which showed the dominancy of either kaolinite or bentonite in these suspensions. This transition was even observed for samples having a high solid content because of the absence of inertial problems in these tests, in contrast to amplitude sweep tests. These results confirmed the existence

of two mechanisms for yielding in mixed suspensions, i.e., breakage of network formed by interacting montmorillonite particles (first yield point) as shown in Fig. S14 and reorganization of non-interacting kaolinite particles (second yield point) as shown in Fig. S15. The yield stress (determined from the viscosity decline and second yield point in case of systems with two-step yielding) is then plotted as a function of kaolinite/bentonite fraction for different total solid content (Fig. 6b). The trend for the yield stress was similar to the cross-over stress and complex modulus at 1 Hz as a function of kaolinite/bentonite fraction, which showed an increase as a function of increasing bentonite fraction. Furthermore, the effect of ionic strength on the yield stress of the sample having lowest bentonite content (8 wt%), as observed for complex modulus, was not very significant. More rheological results for stress ramp-up tests can be found in supplementary information (Fig. S16).

3.3.4. Structural recovery tests

The understanding of structural recovery after pre-shearing is important to analyse the stability of a system, particularly for processes where multiple shearing cycles are involved. The structural recovery
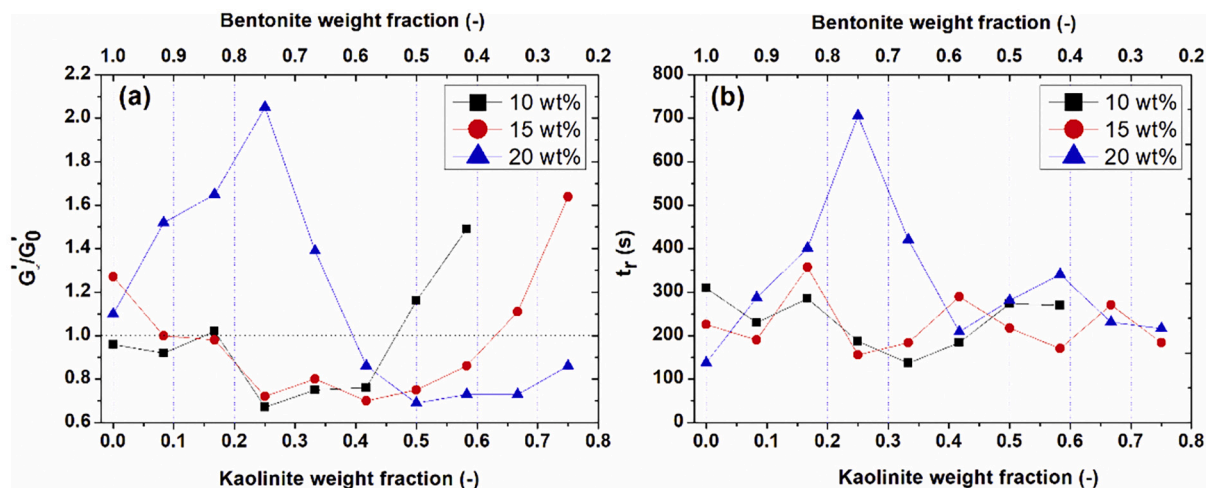
Fig. 7. (a) Equilibrium structure (G_∞'/G_0') and (b) recovery time (t_r) as a function of kaolinite/bentonite ratio for different total solid content. Solid line is just a guide for the eye. The dotted horizontal line represents the structure similar to the one before pre-shearing.

behaviour was studied for mixed suspensions, by varying total solid content and kaolinite/bentonite ratio, using the experimental protocol mentioned in Sec. 2.3. The normalized storage modulus (i.e., time dependent modulus divided by initial modulus) is plotted as a function of time in Fig. S17 for different total solid content and varying kaolinite/bentonite ratios. It can be seen that the samples having higher kaolinite fractions displayed smooth moduli curves as a function of time while for suspensions having higher bentonite fractions, an oscillating behaviour was observed for the moduli curves. This can be linked to the solid-like character or the elasticity of the samples (Goudoulas and Germann, 2016; Shakeel et al., 2020a). These results verified the existence of a strong network of particles at high bentonite fractions.

In order to further quantify the structural recovery behaviour, two parameters (i.e., G_∞'/G_0' and t_r) are plotted as a function of kaolinite/bentonite fraction for different total solid content (see Fig. 7a and b). For low solid content (10–15 wt%), a kind of minimum was observed for the parameter (G_∞'/G_0') as a function of kaolinite/bentonite fraction (Fig. 7a). This showed that for intermediate kaolinite/bentonite fractions, where two-step yielding was found, the structural recovery was lowest which may be linked to the existence of two mechanisms for yielding (Shakeel et al., 2020a). The recovery time (t_r) as a function of kaolinite/bentonite fraction, for low solid content (10–15 wt%), fluctuated around the value of 250 s (Fig. 7b). For higher solid content (20 wt%), a maximum in (G_∞'/G_0') was found instead of a minimum, for intermediate kaolinite/bentonite ratios. Likewise, a maximum was also observed in recovery time for intermediate kaolinite/bentonite fractions, at higher solid content. More rheological results for structural recovery tests can be found in supplementary information (Fig. S17). The values of the fitting parameters found using (Eq. 1) for mixed suspensions with varying solid content and kaolinite/bentonite fractions are presented Table S8.

3.3.5. Multiple shearing cycles

In order to analyse the effect of multiple shearing cycles on the structural recovery of mixed suspensions, multiple structural recovery tests were performed on a same sample (total solid content = 15 wt%; kaolinite/bentonite ratio = 75/25 wt%). This sample was selected on the basis of settling column experiments, presented in the next section, which showed the stability of this sample for long time. The outcome of multiple shearing tests and the structural recovery parameters are shown in Fig. S18a and S18b. It can be clearly seen from Fig. S18b that (G_∞'/G_0') was almost the same even after 10 shearing cycles while the recovery time (t_r) showed an increasing trend as function of shearing cycle. In other words, this behaviour demonstrated that the strength of

Table 3
Bentonite content needed to stabilize the suspension for different total solid content.

Total solid content (wt%)	Bentonite content for stabilization (wt%)
5	2.1
10	3.3
15	3.8
20	3.4

stable mixed suspensions remained the same even after multiple shearing cycles, however, it required longer times to obtain that level of structure after multiple shearing cycles. The values of the fitting parameters found using (Eq. 1) for mixed suspensions with multiple shearing cycles are presented Table S9.

3.3.6. Settling column tests

In addition to the detailed rheological analysis, settling column analysis was also performed for the mixed suspensions. The pictures of settling columns having different total solid content and varying kaolinite/bentonite fractions are shown in Fig. S19-S21 for different time intervals. Fig. S19 shows that the sample containing kaolinite displayed significant settling of particles even after 1 h. However, sample having 67/33 wt% of kaolinite/bentonite showed almost no settling of particles, even after 48 h. The corresponding clay/water interface height as a function of time is also plotted, see Fig. S22. It was found that a very small amount of bentonite was needed to stabilize the kaolinite suspensions (Table 3), without adding any significant amount of salt or polymer. This result verified that the sample having no yield stress showed significant settling behaviour while the stabilized suspension displayed the existence of a yield stress.

4. Conclusions

Bentonite suspensions display high values of viscosity and yield stresses, even with lower content of solids, due to the swelling nature of montmorillonite present in bentonite. In contrast, kaolinite suspensions exhibit non-Newtonian behaviour (i.e., yield stress) at high solid content only, due to its non-swelling nature. The rheological behaviour of mixed kaolinite/bentonite suspensions can be tuned by mixing them in different ratios. In this study, the yielding transitions in mixed kaolinite/bentonite suspensions have been investigated by varying the kaolinite/bentonite ratio and the total solid content. Amplitude sweep tests, frequency sweep tests, stress ramp-up tests and structural recovery tests
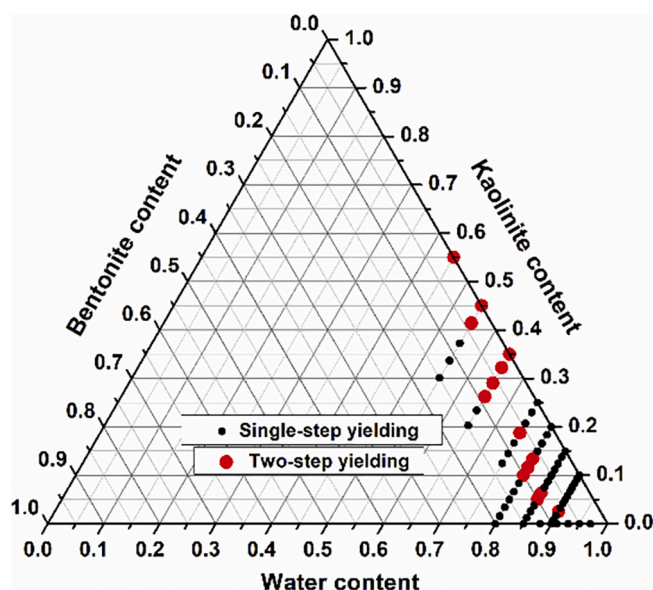
Fig. 8. Ternary diagram for water/kaolinite/bentonite suspensions, black dots represent systems with single-step yielding and red dots represent systems having two-step yielding behaviour. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

were performed for the individual and mixed clay suspensions. All the suspensions were prepared using deionized water (i.e., very low salinity).

The results of stress and amplitude sweep tests showed that the kaolinite suspensions exhibited a two-step yielding behaviour at high solid content (i.e., $\geq$ 35 wt%), a single-step yielding behaviour at intermediate fractions (i.e., 15 wt% $< \phi <$ 35 wt%) and no yield stress for low kaolinite content <15 wt%. On the contrary, bentonite suspensions displayed a single-step yielding behaviour for all the investigated clay concentrations (i.e., 3–20 wt%). This behaviour may be associated to the strong electrostatically repulsive forces between montmorillonite particles at low ionic strength. In mixed kaolinite/bentonite suspensions, a clear transition between a single-step and two-step yielding was observed in steady and oscillatory tests as a function of kaolinite/bentonite ratio, for a particular total solid content. Fig. 8 summarizes the yielding transitions in individual and mixed clay suspensions. Frequency sweep and stress ramp-up tests showed that the moduli and yield stress of mixed suspensions can be tuned by varying the kaolinite/bentonite ratio and the total solid content. An ionic strength dependence was observed for the mixed kaolinite/bentonite suspensions in frequency sweep tests at small amounts of bentonite. However, this dependence was not significantly affecting the yield stress values of the suspensions. Further systematic analysis is needed to study the influence of ion type and concentration on the rheological properties of mixed kaolinite/bentonite suspensions.

The structural recovery experiments showed that for mixed kaolinite/bentonite suspensions having intermediate kaolinite/bentonite ratios, the structural recovery was lowest at low total solid content (10–15 wt%) while at higher solid content (20 wt%) the recovery was highest, which may be linked to the existence of a two-step yielding. Furthermore, the outcome of structural recovery tests for stable kaolinite/bentonite suspensions displayed that the structure (i.e., moduli) of the suspensions was completely recovered to its initial state, even after multiple shearing cycles. The settling column analysis displayed that a very small amount of bentonite (2–4 wt%) was needed to prevent the settling of kaolinite suspensions. This study could be very useful for the applications where the settling of clay particles in suspensions is not required, such as, for producing stable commercial

formulations based on clay. Moreover, this study also highlights the influence of even small amount of bentonite on the dewatering behaviour of kaolinite suspensions along with its rheological fingerprint, which can provide useful insights for the processing and disposal of clay-rich oil sand tailings.

Credit author statement

Ahmad Shakeel: Methodology, Investigation, Formal analysis, Writing – Original Draft.

Alex Kirichek: Conceptualization, Supervision, Writing – Review & Editing.

Claire Chassagne: Funding acquisition, Supervision, Writing – Review & Editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.clay.2021.106206.

References

Abend, S., Lagaly, G., 2000. Sol–gel transitions of sodium montmorillonite dispersions. Appl. Clay Sci. 16, 201–227.

Adebayo, T.A., Ajayi, O., 2011. Unprocessed OTA kaolin as a weighting additive in drilling fluid. Asian Transac. on Eng. 1, 23–26.

Ahuja, A., Gamonpilas, C., 2017. Dual yielding in capillary suspensions. Rheol. Acta 56, 801–810.

Au, P.-I., Leong, Y.-K., 2013. Rheological and zeta potential behaviour of kaolin and bentonite composite slurries. Colloids Surf. A Physicochem. Eng. Asp. 436, 530–541.

Barnes, H.A., 1995. A review of the slip (wall depletion) of polymer solutions, emulsions and particle suspensions in viscometers: its cause, character, and cure. J. Non-Newtonian Fluid Mech. 56, 221–251.

Chalaturnyk, R.J., Don Scott, J., Özüm, B., 2002. Management of oil sands tailings. Pet. Sci. Technol. 20, 1025–1046.

Chang, C., Powell, R.L., 1994. Effect of particle size distributions on the rheology of concentrated bimodal suspensions. J. Rheol. 38, 85–98.

Choo, K.Y., Bai, K., 2015. Effects of bentonite concentration and solution pH on the rheological properties and long-term stabilities of bentonite suspensions. Appl. Clay Sci. 108, 182–190.

Coussot, P., 1997. Mudflow Rheology and Dynamics. Routledge.

Czibulya, Z., Tombácz, E., Szegi, T., Michéli, E., Zsolnay, Á., 2010. Standard state of soil dispersions for rheological measurements. Appl. Clay Sci. 48, 594–601.

Dagois-Bohy, S., Somfai, E., Tighe, B.P., van Hecke, M., 2017. Softening and yielding of soft glassy materials. Soft Matter 13, 9036–9045.

Daniel, W.E., Eibling, R.E., 2005. Rheology Modifiers Applied to Kaolin-Bentonite Slurries for SRNL WTP Pulse Jets Tank Pilot Work in Support of RPP at Hanford, pp. 1–12.

Goh, R., Leong, Y.-K., Lehane, B., 2011. Bentonite slurries—zeta potential, yield stress, adsorbed additive and time-dependent behaviour. Rheol. Acta 50, 29–38.

Goudoulas, T.B., Germann, N., 2016. Viscoelastic properties of polyacrylamide solutions from creep ringing data. J. Rheol. 60, 491–502.

Grabowska-Olszewska, B., 2003. Modelling physical properties of mixtures of clays: example of a two-component mixture of kaolinite and montmorillonite. Appl. Clay Sci. 22, 251–259.

Heymann, L., Peukert, S., Aksel, N., 2002. Investigation of the solid–liquid transition of highly concentrated suspensions in oscillatory amplitude sweeps. J. Rheol. 46, 93–112.

Huang, W., Leong, Y.-K., Chen, T., Au, P.-I., Liu, X., Qiu, Z., 2016. Surface chemistry and rheological properties of API bentonite drilling fluid: pH effect, yield stress, zeta potential and ageing behaviour. J. Pet. Sci. Eng. 146, 561–569.

Ibanez Sanz, M., 2018. Flocculation and Consolidation of Cohesive Sediments under the Influence of Coagulant and Flocculant. Delft University of Technology.

İşci, S., Günister, E., Ece, Ö.I., Güngör, N., 2004. The modification of rheologic properties of clays with PVA effect. Mater. Lett. 58, 1975–1978.

Johnson, S.B., Dixon, D.R., Scales, P.J., 1999. The electrokinetic and shear yield stress properties of kaolinite in the presence of aluminium ions. Colloids Surf. A Physicochem. Eng. Asp. 146, 281–291.

Kasperski, K.L., Hepler, C.T., Hepler, L.G., 1986. Viscosities of dilute aqueous suspensions of montmorillonite and kaolinite clays. Can. J. Chem. 64, 1919–1924.

Kelessidis, V.C., Maglione, R., 2008. Yield stress of water–bentonite dispersions. Colloids Surf. A Physicochem. Eng. Asp. 318, 217–226.

Kelessidis, V.C., Tsamantaki, C., Dalamarinis, P., 2007. Effect of pH and electrolyte on the rheology of aqueous Wyoming bentonite dispersions. Appl. Clay Sci. 38, 86–96.

Keren, R., 1989. Rheology of mixed Kaolinite-Montmorillonite Suspensions. Soil Sci. Soc. Am. J. 53, 725–730.

Kiviranta, L., Kumpulainen, S., 2011. Quality control and characterization of bentonite materials (no. POSIVA-WR–11-84). Posiva Oy.

Lagaly, G., 1989. Principles of flow of kaolin and bentonite dispersions. Appl. Clay Sci. 4, 105–123.

Laird, D.A., 2006. Influence of layer charge on swelling of smectites. Appl. Clay Sci. 34, 74–87.

Läuger, J., Stettin, H., 2016. Effects of instrument and fluid inertia in oscillatory shear in rotational rheometers. J. Rheol. 60, 393–406.

Lee, C.E., Chandra, S., Leong, Y.-K., 2012. Structural recovery behaviour of kaolin, bentonite and K-montmorillonite slurries. Powder Technol. 223, 105–109.

Leong, Y.-K., Scales, P.J., Healy, T.W., Boger, D.V., 1995. Effect of Particle size on Colloidal Zirconia Rheology at the Isoelectric Point. J. Am. Ceram. Soc. 78, 2209–2212.

Leong, Y.-K., Teo, J., Teh, E., Smith, J., Widjaja, J., Lee, J.-X., Fourie, A., Fahey, M., Chen, R., 2012. Controlling attractive interparticle forces via small anionic and cationic additives in kaolin clay slurries. Chem. Eng. Res. Des. 90, 658–666.

Lin, Y., Phan-Thien, N., Lee, J.B.P., Khoo, B.C., 2015. Concentration Dependence of Yield stress and Dynamic Moduli of Kaolinite Suspensions. Langmuir 31, 4791–4797.

Luckham, P.F., Rossi, S., 1999. The colloidal and rheological properties of bentonite suspensions. Adv. Colloid Interf. Sci. 82, 43–92.

Luckham, P.F., Ukeje, M.A., 1999. Effect of Particle size distribution on the Rheology of Dispersed Systems. J. Colloid Interface Sci. 220, 347–356.

Melton, I.E., Rand, B., 1977. Particle interactions in aqueous kaolinite suspensions: II. Comparison of some laboratory and commercial kaolinite samples. J. Colloid Interface Sci. 60, 321–330.

Mobuchon, C., Carreau, P.J., Heuzey, M.-C., 2007. Effect of flow history on the structure of a non-polar polymer/clay nanocomposite model system. Rheol. Acta 46, 1045–1056.

Mobuchon, C., Carreau, P.J., Heuzey, M.-C., 2009. Structural analysis of non-aqueous layered silicate suspensions subjected to shear flow. J. Rheol. 53, 1025–1048.

Montes-H, G., Duplay, J., Martinez, L., Geraud, Y., Rousset-Tournier, B., 2003. Influence of interlayer cations on the water sorption and swelling–shrinkage of MX80 bentonite. Appl. Clay Sci. 23, 309–321.

Mpofu, P., Addai-Mensah, J., Ralston, J., 2003. Influence of hydrolyzable metal ions on the interfacial chemistry, particle interactions, and dewatering behavior of kaolinite dispersions. J. Colloid Interface Sci. 261, 349–359.

Mpofu, P., Addai-Mensah, J., Ralston, J., 2004. Flocculation and dewatering behaviour of smectite dispersions: effect of polymer structure type. Miner. Eng. 17, 411–423.

Palomino, A.M., Santamarina, J.C., 2005. Fabric Map for Kaolinite: Effects of Ph and Ionic Concentration on Behavior. Clay Clay Miner. 53, 211–223.

Prasad, M.S., Reid, K.J., Murray, H.H., 1991. Kaolin: processing, properties and applications. Appl. Clay Sci. 6, 87–119.

Sentjabrskaja, T., Babaliari, E., Hendricks, J., Laurati, M., Petekidis, G., Egelhaaf, S.U., 2013. Yielding of binary colloidal glasses. Soft Matter 9, 4524–4533.

Shakeel, A., Kirichek, A., Chassagne, C., 2019a. Is density enough to predict the rheology of natural sediments? Geo-Mar. Lett. 39, 427–434.

Shakeel, A., Mahmood, H., Farooq, U., Ullah, Z., Yasin, S., Iqbal, T., Chassagne, C., Moniruzzaman, M., 2019b. Rheology of Pure Ionic Liquids and their complex Fluids: a Review. ACS Sustain. Chem. Eng. 7, 13586–13626.

Shakeel, A., Kirichek, A., Chassagne, C., 2020a. Effect of pre-shearing on the steady and dynamic rheological properties of mud sediments. Mar. Pet. Geol. 116, 104338.

Shakeel, A., Kirichek, A., Chassagne, C., 2020b. Rheological analysis of mud from Port of Hamburg, Germany. J. Soils Sediments 20, 2553–2562.

Shakeel, A., Kirichek, A., Chassagne, C., 2020c. Rheological analysis of natural and diluted mud suspensions. J. Non-Newtonian Fluid Mech. 286, 104434.

Shakeel, A., Kirichek, A., Chassagne, C., 2020d. Yield stress measurements of mud sediments using different rheological methods and geometries: an evidence of two-step yielding. Mar. Geol. 427, 106247.

Shakeel, A., Safar, Z., Ibanez, M., van Paassen, L., Chassagne, C., 2020e. Flocculation of Clay Suspensions by Anionic and Cationic Polyelectrolytes: a Systematic Analysis. Minerals 10, 999.

Teh, E.J., Leong, Y.K., Liu, Y., Fourie, A.B., Fahey, M., 2009. Differences in the rheology and surface chemistry of kaolin clay slurries: the source of the variations. Chem. Eng. Sci. 64, 3817–3825.

Werner, B., Myrseth, V., Saasen, A., 2017. Viscoelastic properties of drilling fluids and their influence on cuttings transport. J. Pet. Sci. Eng. 156, 845–851.

Yalçın, T., Alemdar, A., Ece, Ö.I., Güngör, N., 2002. The viscosity and zeta potential of bentonite dispersions in presence of anionic surfactants. Mater. Lett. 57, 420–424.

Zhao, C., Yuan, G., Han, C.C., 2014. Bridging and caging in mixed suspensions of microsphere and adsorptive microgel. Soft Matter 10, 8905–8912.