Yield Stress of Emulsions and Suspensions as Measured in Steady Shearing and in Oscillations

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Abstract:
The yield stresses of five samples (two highly concentrated emulsions, two Kaolin dispersions and mayonnaise) were determined in two ways. In one case, steady shear experiments were performed over a range of incrementally decreasing shear rates. The resulting flow curves, plotted as shear stress against shear rate, clearly showed the existence of a yield stress for each sample, the Herschel-Bulkley model being fitted to obtain values. In the second case, oscillatory amplitude sweeps were performed at three frequencies, and the "dynamic yield stress" was defined as the stress at which deviation from linearity occurred; this procedure has often been used to determine the yield stress of emulsions. It was found that the dynamic yield stress is frequency dependent, and cannot therefore be thought of as physically meaningful material property. At no frequency did the dynamic yield stress correlate with the yield stress obtained from the flow curves.

Zusammenfassung:
Die Fließgrenzen von fünf Proben (zwei hochkonzentrierte Emulsionen, zwei Kaolin-Dispersionen und Mayonnaise) wurden auf zweierlei Arten ermittelt. In einem Falle wurden stetige Scherversuche über eine Reihe von stetig abnehmenden Scherraten durchgeführt. Die sich ergebenden Fließkurven, dargestellt als Fließgrenze gegenüber der Scherrate, zeigten klar das Bestehen einer Fließgrenze für jede Probe, wobei das Herschel-Bulkley-Modell zur Errechnung der Werte angepasst wurde. Im zweiten Falle wurden oszillierende Amplitudensweeps bei drei Frequenzen durchgeführt, wobei die „dynamische Fließgrenze" definiert wurde als die Spannung, bei der eine Abweichung von der Linearität auftrat. Dieses Verfahren wurde oft für die Ermittlung der Fließgrenze von Emulsionen verwendet. Es wurde festgestellt, dass die dynamische Fließgrenze frequenzabhängig ist und daher nicht als sinnvolle Materialeigenschaft angesehen werden kann. Bei keiner Frequenz korrelierte die dynamische Fließgrenze mit der aus den Fließkurven hervorgehenden Fließgrenze.

Résumé:
La contrainte d'écoulement de cinq échantillons (deux émulsions à forte concentration, deux dispersions au Kaolin et une mayonnaise) a été déterminée de deux façons. Dans un cas, les expériences de cisaillement continu ont été effectuées sur une gamme de taux de cisaillement décroissants. Les courbes d'écoulement résultantes, tracées comme contrainte de cisaillement par rapport au taux de cisaillement, montrent clairement l'existence d'une contrainte d'écoulement dans chaque échantillon, le modèle Herschel-Burkley ayant été utilisé pour obtenir les valeurs. Dans le second cas on a effectué un balayage d'amplitude oscillatoire sur trois fréquences, et la « contrainte dynamique d'écoulement » a été définie comme la contrainte à laquelle avait lieu la déviation de la linéarité ; cette procédure a souvent été utilisée pour déterminer la contrainte d'écoulement des émulsions. On a trouvé que la contrainte dynamique d'écoulement dépend de la fréquence, et par conséquent ne peut pas être considérée comme une propriété physiquement signifiante du matériel. Sous aucune fréquence la contrainte dynamique d'écoulement a été en corrélation avec la contrainte d'écoulement obtenue des courbes d'écoulement.

Key words: yield stress, emulsions, kaolin, mayonnaise, flow curves, amplitude sweep

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1 INTRODUCTION

The behaviour of different materials at very low shear stresses has always been a subject of special interest. This interest arises from the idea that applying low stresses allows the properties of the unperturbed structure of a material to be explored.

Generally speaking, there are two limiting situations. In one case a regime of constant viscosity is observed over a relatively wide range of shear rates (or stresses). This viscosity being referred to as the initial Newtonian viscosity, and can be directly related to the molecular mass of a polymer chain [1], the concentration of a solid phase in a dispersion, and so on [2]. In the second case, an unlimited growth in viscosity occurs at some limiting shear stress, below which flow becomes impossible. This may be viewed as a transition to solid-like behaviour. If the transition takes place sharply, the transition point is deemed to be the yield stress. The phenomenon of the yield stress was proposed as the first milestone of rheology [3] and continues to form the focus of numerous authors. The concept of yielding at low stresses is commonly accepted for many materials, including various different types of suspension [4 - 7], consistent greases [8], filled polymer melts [9], and ice suspensions [10], to name but some. The existence of a yield stress was also quite evident in our studies of the rheology of highly concentrated emulsions [11 - 16], as well as occurring in other emulsions such as mayonnaise [17, 18]. The parameter can be used for the quantitative comparison of different materials. In particular, it has been shown that the yield stress directly reflects the evolution of the physical structure of emulsions in time [14].

However, in the determination of either the low-shear-rate Newtonian viscosity or the yield stress, some doubt remains: are the experimental conditions appropriate to speak about a genuinely threshold situation? This problem has been discussed in several publications [19 - 22]. As the yield stress is often found by extrapolation of experimental data, the result may strongly depend on the chosen procedure of experimental data fitting. So the conclusion: “It depends on what you mean ... by the yields stress” [22] seems to be the most reasonable answer to the problem of measuring “the yield stress”.

But at least in some cases, the existence of yielding and the transition from solid-like to liquid-like behaviour seem evident. This is true for many multi-component systems where a disperse phase creates some form of rigid structure and the yield stress characterizes its strength. Besides the experimental evidences, the existence of a physically-based yielding behaviour of highly concentrated emulsions was proven in several theoretical publications devoted to modelling the properties of these materials [23 - 26].

Nevertheless, the problem of the experimental determination of the yield stress continued to be debated. As a general rule, the yield stress is assigned to the vertical part of a viscosity against shear stress curve obtained from different deformation modes, and particularly from the decreasing shear rate mode. In that case, the value of the yield stress is either quite evident and unambiguous or is found by extrapolation of experimental points, using a fitting equation such as the Herschel-Bulkley model [2]. Moreover, several authors have attributed the non-linearity observed in the viscoelastic properties of different materials as the consequence of yielding. Kamatsu et al. [27] treated the distortion of the stress waves from the sinusoidal shape as the “dynamic yield stress”. Mason et al. [25] and Babak et al. [28] treated the initial point of non-linearity in the amplitude dependence of the elastic modulus as the physically meaningful yield stress and used this value in their theoretical speculations.

In discussing the rheological properties of multi-component systems (suspensions and emulsions), it is necessary to pay special attention to the wall slip phenomenon and to exclude this effect before discussing the true bulk properties of a material and the yield stress in particular. Slip occurs in the flow of multi-phase systems because of the displacement of the disperse phase away from solid boundaries. This arises from steric, hydrodynamic, viscoelastic and chemical forces and the constraints acting on the disperse phase immediately adjacent to the walls. The slip effect was reported to exist in suspension and emulsion systems by Barnes [29], and specifically in kaolin suspensions [30, 31] and mayonnaise [32, 33].

The goal of this note is to compare the results of two different methods of estimating the yield stress – in steady shearing and in peri-

Mineralogical composition	Kaolinite (race mica & quartz)
Abrasiveness	25 g/m²
Particle size distribution	< 20 µm: 98 % < 10 µm: 90 % < 2 µm: 48 %
Mean particle size	2.1 µm
BET surface area	18.73 m²/g
pH value	4.0 ~ 5.0
Specific gravity	2.6
Mohs hardness	2.0 ~ 2.5

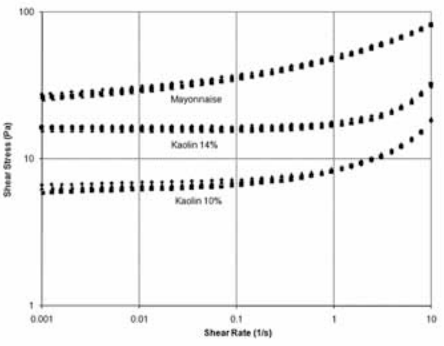


Table 1 (left): Kaolin characteristics.

Figure 1 (right): The flow curve of Kaolin-10, Kaolin-14 and mayonnaise sample obtained by parallel-plate geometry with different gaps (◇: 0.8 mm, □: 1 mm, △: 1.2 mm).

odic oscillation. Though the main subject of our work is highly concentrated emulsions, the problem was studied using a wider range of materials: kaolin at two concentrations in water as a typical suspension, and mayonnaise as a typical emulsion different from liquid explosives. This was done to make the conclusions of comparison of the two methods under discussion more reliable.

2 EXPERIMENTAL

2.1 MATERIALS

Highly concentrated emulsions of the explosive type. These materials were described in detail in our earlier publications [11 - 16, 34]. Consequently only some of principle features need to be repeated here. The samples are emulsions of the water-in-oil type with a concentration of the aqueous phase up to 96 wt%. The liquid droplets have a polygonal shape and consist in a supercooled aqueous solution. Water comprises less than 20 % by mass of this phase, the remainder being mainly inorganic salts. The oil phase is based on hydrocarbons and the emulsifier comprises approximately 15 % of this phase. These emulsions are visco-plastic materials with strong non-Newtonian behaviour at stresses exceeding some limit treated as the yield stress. The transition to the vertical part of the viscosity against stress curves – to yielding – is quite well visible and evident.

In this study, two typical examples of explosive emulsions were used. These samples, designated as D-13 and D-14, are similar in their chemical composition. They contain 6 wt% oil as the continuous phase. The average droplet size is 13 and 14 μm, respectively. The size distribution of droplet size is Gaussian [16, 33]. The yielding behaviour of these systems is dependent on the structure formed by surface inter-droplet layers of surfactant and, possibly, on the direct contact of droplets. The latter supposition is based on the observation that the inner structure of droplets (which changes as the ammonium nitrate crystallizes) directly influences the yield stress [14].

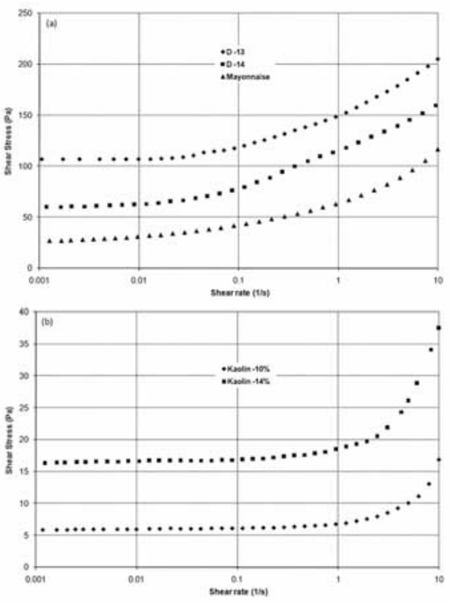
Kaolin dispersions. The characteristics of the kaolin powder used for experiments are listed in Table 1. Two water dispersions with different concentrations of kaolin - 10 and 14 % (designated below as Kaolin-10 and Kaolin-14, respectively) were prepared by a 30 minute agitation of the water and kaolin mixtures. The yielding behaviour of these suspensions is dependent on the mechanical contacts of solid particles creating a structure with its own strength.

Mayonnaise. The mayonnaise sample is manufactured by Kraft Foods South Africa Ltd., and marketed under the tradename "REAL". The ingredients are listed as: 52 % sunflower oil, water, modified starch, vinegar, pasteurized egg, sugar, salt, preservatives, lemon juice, thickener, garlic and onion powder and spices. The yielding behaviour of this emulsion is assumed to be due the formation of a network structure by lipoproteins [17].

2.2 METHODS

Samples were tested using a rotational rheometer MCR300 (Paar Physica) with a cup and bob measuring system. A bob with a sandblasted surface was used. The possibility of wall slip in our experiments was investigated using a plate-plate (sandblasted-smooth) geometry at different gap sizes. The wall roughness of these two geometries, cup and bob and plate-plate, was similar, the two systems therefore being analogous to each other. The diameter of the sandblasted plate was 50 mm.

Experiments were carried out using various deformation regimes. Firstly, stress-controlled deformations were applied, the stress being changed incrementally. Secondly, flow curves were measured in a downward shear rate sweeping mode as described in earlier publications



Sample	Fitting Range [s^{-1}]	σ_Y*	*K*	*n*
D -13	0.001 ~ 0.02	107.21	12158.65	2.66
D -14	0.001 ~ 0.02	59.36	130.82	0.80
Mayonnaise	0.001 ~ 1	21.56	42.87	0.32
Kaolin 10%	0.001 ~ 5	6.02	0.81	1.01
Kaolin 14%	0.001 ~ 3	16.51	2.08	0.74

Figure 2 (left): The flow curves of (a) three different emulsions (two explosive and a mayonnaise) and (b) two different suspensions.

Table 2 (right): Values of Herschel-Bulkley model fitted on different samples.

[13, 15]. Two different time scales, 5 and 10 s, were used for each experimental point of the flow curves for all samples. It is worth noting that the flow curves of all samples were independent of the shearing ramp chosen. The procedure of estimating the yield stress will be discussed in the following section of the paper. Thirdly, the amplitude dependencies of the dynamic moduli were measured at several constant frequencies 1, 10 and 100 rad/s. For highly concentrated emulsions, the storage modulus does not depend on frequency, and the frequency dependence of the loss modulus is rather weak. However, it was not evident beforehand whether the transition from linear to non-linear amplitude dependence was sensitive to frequency.

The results are presented below as the amplitude dependencies of the real (storage) and imaginary (loss) moduli obtained from strain sweep or stress sweep experiments. The yield stress was estimated from the amplitude dependence of the stress or strain in the manner discussed below.

3 RESULTS AND DISCUSSION

Before discussing the experimental results related to the bulk flow properties of the materials under study including the problem of the correct determination of the yield stress, it is necessary to eliminate the possible influence of the wall slip phenomenon.

The problem of slip in measuring the flow properties of highly filled systems always exists and should be taken into account. This problem was specially studied for explosive emulsions in our earlier publications [13 - 15]. Two types of experiments were carried out: flow through tubes (including industrial pipe-lines) of different diameter, and rotational flow using devices with different gap between stationary smooth and rotating sandblasted surfaces. In all cases, no influence of wall slip on the results of rheological measurements and output calculations for a pipe-line was established. So the results were invariant with respect to the wall/volume ratio. It means that we can neglect this possible phenomenon and treat the results of rheological measurements for these systems as the true bulk properties of the materials.

For kaolin suspensions, two types of experiment were also carried out. The first one is the flow through tubes of different diameter. In an earlier publication [35], we experimented with the pipe flow of the kaolin suspensions through tubes of 13, 28, and 80 mm diameter, and compared the technological data with predictions based on laboratory viscometric measurements of these suspensions carried out using a rotational rheometer with sandblasted surfaces. The results showed no evidence for the existence of any slip effect. Furthermore, experiments with different gap sizes were performed in the present work to investigate the possibility of slip in the rheological results of Kaolin-10, Kaolin-14 and mayonnaise samples. This was carried out using the parallel-plate geometry with sandblasted surfaces, as discussed in the experimental section. The results of measurements clearly show (Fig. 1) that the experimental technique

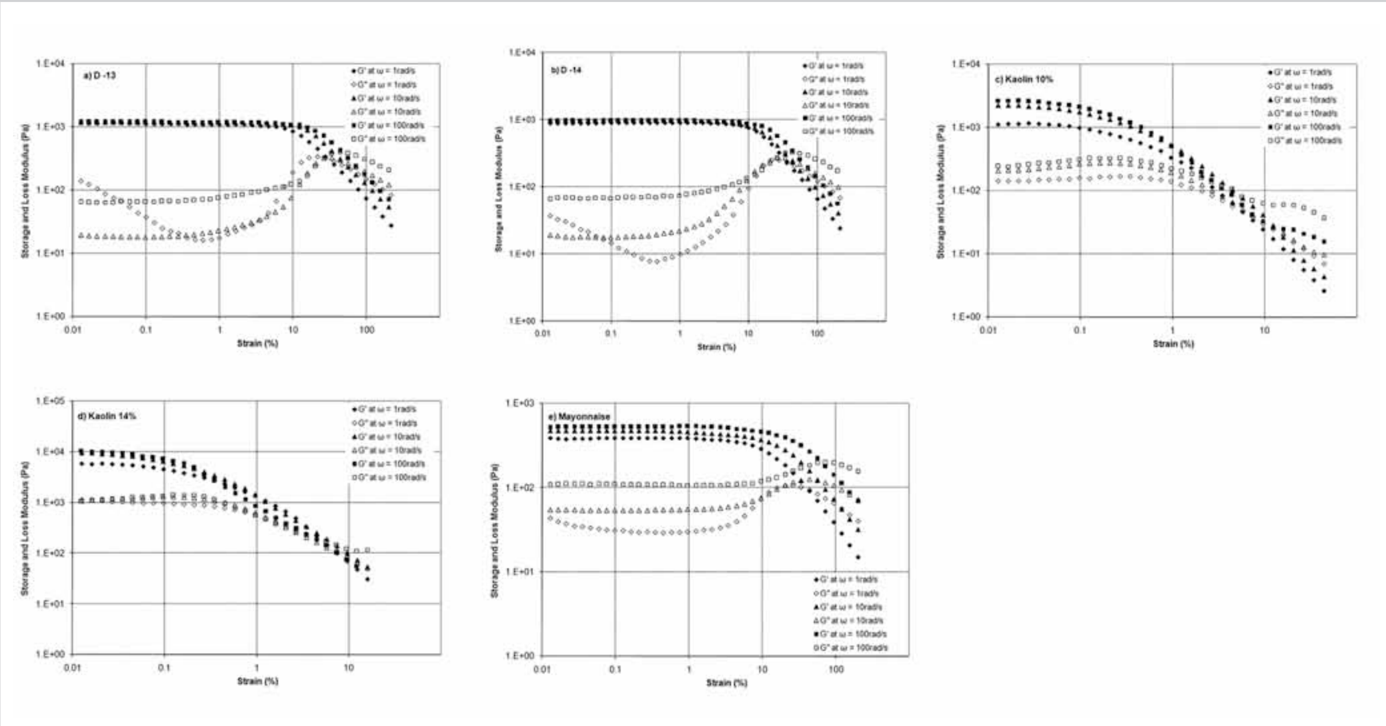


Figure 3 (above): Results of amplitude sweep experiments at three angular frequencies for different samples.

Table 3 (below): Summary of the experimentally obtained values of characteristic stresses.

Sample	σ_Y^*	σ^1	σ^{10}	σ^{100}
D -13	107.21	118.2	139.2	170.8
D -14	58.36	99.7	102.9	136.9
Mayonnaise	6.02	0.8	1.2	1.4
Kaolin 10%	16.51	3.0	3.7	4.0
Kaolin 14%	21.56	38.2	54.4	78.1

All values of stresses are given in Pa

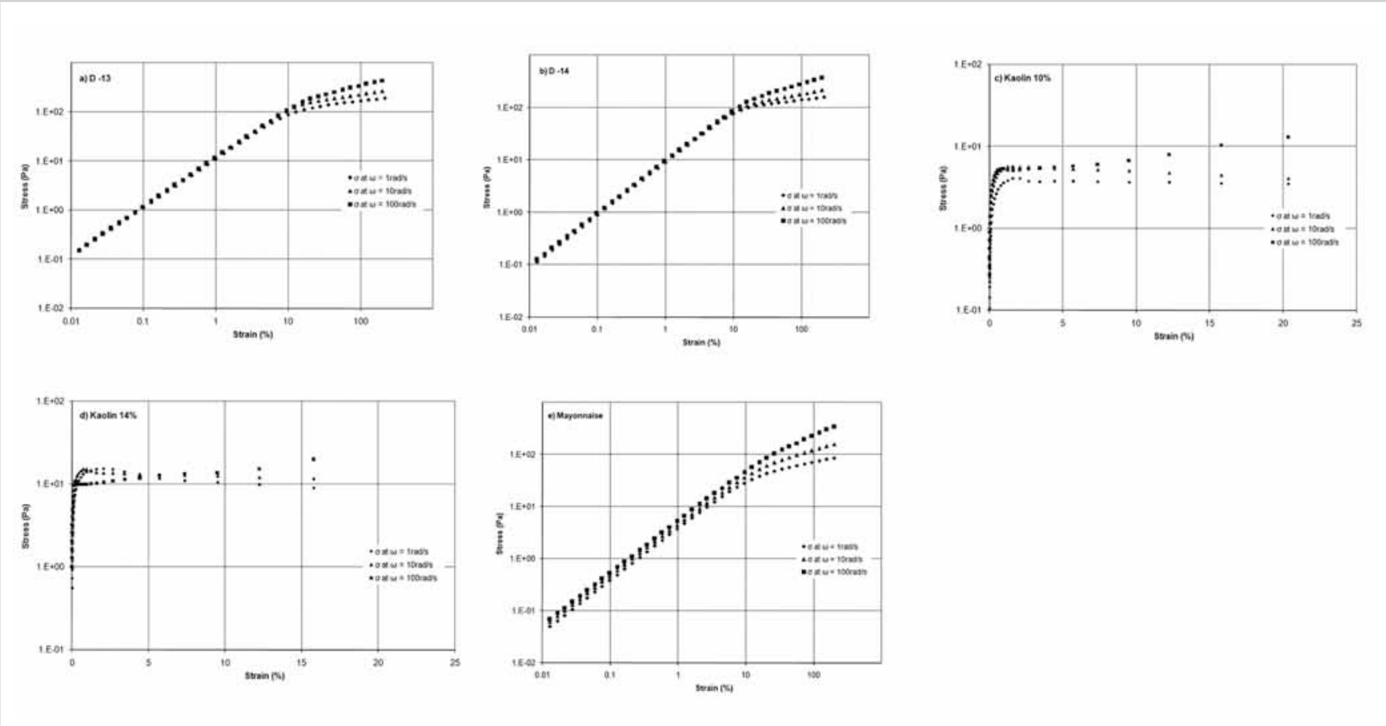
used allows us to assume that the slip can be neglected.

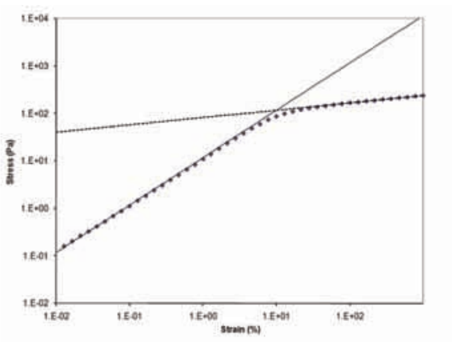
The lower parts (related to the low shear rate domain) of flow curves for the five materials under study are presented in Figure 2a-b. These data were obtained in the downward sweeping shear rate experiments, because it has been shown [13, 15] that the real flow curve can be best obtained in this mode of deformations. The data are presented on semi-log scales in order to demonstrate that the stress level really remains constant over a rather wide range of shear rates. The yield stress was obtained by fitting the Herschel-Bulkley model, $\sigma = \sigma_Y^* + K\dot{\gamma}^n$, to the low shear rate region of flow curve (Table 2), where the yield stress is denoted by σ_Y^*. In all cases a good fit was achieved. Thus the yield stress is a real physical characteristic of a material and the values will be used as the benchmark for comparison with other values of characteristic parameters obtained in different experiments. The error in the estimation of σ_Y^* is no more than 10% and in the most cases is less than 5 %.

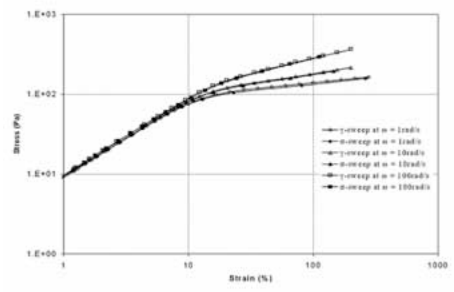
The frequency dependencies of the dynamic moduli are presented in Figure 3a-e for the linear domain of viscoelasticity. One can clearly see the difference in the properties of highly concentrated emulsions, suspensions and a regular emulsion (mayonnaise). While highly concentrated emulsions resemble quasi-solid materials as shown by the absence of any frequency dependence on elastic modulus, other materials demonstrate a strong dependence of the elastic modulus on frequency.

In accordance to the aim of this study, we will estimate the point of deviation of the mechanical properties in the samples under study from the linear regime of viscoelastic behaviour. Several examples of the amplitude of strain sweep are presented in Figure 4 for three types of materials: highly concentrated emulsions (a), suspensions (b) and the regular emulsion (c).

It can be seen that the non-linearity is much more strongly expressed in the loss modulus than in storage modulus. This is quite typical for







many materials. We have chosen the following procedure for estimating the "critical" point - the limit of linear behavior - for highly concentrated emulsions. Following the method proposed by Mason et al. (1996) [25], we plotted the stress amplitude against the strain amplitude and found the critical point from the intersection of straight lines as shown in Figure 5. This method was used for several frequencies and the intersections have been designated as σ^1, σ^{10}, and σ^{100}, where the superscript shows the frequency, at which the intersection stress was measured. The same method was also used for mayonnaise.

However this method does not work for suspensions because their stress against strain curves look different (see Figure 4c, d). In this case, the critical point was assumed as the start of departure from linear dependency of stress against strain curve when the strain increases. The summary of all experimental results is presented in Table 3. At this point, one might suspect that the differences in yield stress are due to the strain sweep mode in the experiments. Therefore the experiments were repeated in the stress sweep mode. Figure 6 demonstrates the results of measurements for one of the samples. One can see that there is no difference between the results of these two modes in linear viscoelastic region measurement. Two things now become rather clear. Firstly, the critical stress found in the oscillating tests (sometimes referred to as the "dynamic yield stress") is not constant but strongly depends on frequency. Secondly, the values of the critical stress found in the oscillating experiment do not coincide with real ("physical") yield stress estimated from flow curves. This means that the "dynamic yield stress" is in no event a physical parameter of a material, and cannot be used for comparison with any theoretical conclusion concerning rheological properties of visco-plastic materials. It is possible to try and extrapolate experimental data for "dynamic yield stress" to "zero" frequency. However, even in this approach, we did not reach a reasonable correlation between $\sigma_\gamma{}^*$ and the "zero-frequency dynamic yield stress".

Figure 4 (above):
Stress vs. strain curve at three angular frequencies for different samples (The data for the suspension samples are presented in semi-log scale for better visibility).

Figure 5 (left below):
Stress versus strain dependence for D-13 sample at angular frequency 1 rad/s. The symbols show the experimental points, while the solid line shows the fitting of Hooke law and the dashed line shows the fitting by the power-law nonlinear relation $\sigma = a\gamma^n$

Figure 6 (right below):
The stress versus strain curve of D-14 sample at different angular frequencies and two different sweeping, stress and strain modes (lines are just for directing eye).

4 CONCLUSION

Different visco-plastic materials – suspensions and emulsions – were studied in steady flow mode over a wide range of shear rates and in oscillatory mode with different amplitudes of stress (and strain) and at different frequencies. Flow curve measurements, which were carried out in the downward sweep mode of shearing, showed the existence of a yield stress which was obtained by the fitting of Herschel-Bulkley model.

It was demonstrated that the critical shear stress point estimated from oscillating regimes of deformation as the interception of two lines (corresponding to linear and non-linear domains) in stress against strain dependence, and sometimes considered as the "dynamic yield stress", depends on frequency and therefore cannot be treated as a point with some physical meaning. There is no correlation between the flow curve yield stress (which really reflects the physics of the visco-plastic nature of materials under study) and the dynamic yield stress, no matter at what frequency the latter has been measured.

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