

Rheological and Texture Analysis Methods for Quantifying Yield Value and Level of Thixotropy

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abstract

The objective of this study was to compare different methods for the assessment of yield stress and level of thixotropy in semisolid emulsions, using three instruments: an air-bearing controlled-stress rheometer, a T-bar viscometer and a texture analyser. The following methods were used to assess the yield stress: a low-rotation T-bar test, a shear stress sweep, a shear rate sweep, an oscillatory stress sweep and an immersion/de-immersion test on the texture analyser. The level of thixotropy was assessed by: a hysteresis loop test, a three-step thixotropy test and a repeated immersion/de-immersion test.

A good linear regression fit was found between increasing polymer concentration and yield stress measured by the T bar test, oscillatory stress sweep and immersion/de-immersion test, while no such fit existed in the cases of the shear rate sweep and shear stress sweep. There was good correlation between the two rheological methods for detecting the level of thixotropy, the hysteresis loop and the three-step thixotropy test.

The most consistent yield value results were obtained using the low-rotation T-bar test and the oscillatory stress sweep test. Under the experimental conditions of this study, both three-step thixotropy and hysteresis loop test have proven reliable in assessing the level of thixotropy. The repeated immersion/de-immersion test could also be employed to provide information on immediate structural recovery.

Introduction

The definition of rheology as the study of the deformation and flow of matter is well known and accepted [1]. Yet, most rheological information in the literature concerns only the flow aspect, obtained using continuous measuring methods. The other aspect, the deformation experienced before the sample starts flowing, is often unreported, possibly because it is considered of more academic than practical value. This study used a combination of the steady (continuous) flow and dynamic (oscillatory) methods, aiming to assess their applicability to practical formulation work.

A large number of rheological methods could be found in the literature (e.g. 2), each with a number of variations. However, there is little practical information for cosmetic formulators on the comparative value of different methods used to assess the same parameter, and on their applicability to different laboratory situations. In this study, a comparative assessment of the methods used to establish common rheological parameters was carried out. This work does not cover viscosity, the best known and most ubiquitous rheological parameter. Instead, it focuses on the two rheological parameters of great importance in practical formulation work – yield stress and the level of thixotropy. Both are recognised as useful in understanding how materials behave in the laboratory, in production, during storage and in use [1,2]. It is also well recognised that they are related to the sensory properties of cosmetic emulsions [3-7], whereby the initial touch is often associated with the yield stress and spreadability with thixotropy.

The standard definition of yield stress (yield value or yield point) is the shear stress below which no flow can be observed under the conditions of experimentation [8]. It is also called static yield stress, as opposed to dynamic, which defines the stress needed to maintain the flow [8]. Below yield stress, the material undergoes deformation, while above yield stress it flows, which means that higher yield stress values indicate higher level of elasticity within a viscoelastic material. However, this is a simplified approach to yield stress, adopted for practical reasons. The yield of any semisolid structure is a region, not a point. This means that the reported yield value depends on the way used to determine the point of interest within a given method. Together with a variety of methods in existence, this makes it almost impossible to compare the values found in the literature.

Thixotropy is one of the most complex rheological phenomena in colloid science, which is reflected in the multitude of definitions in the scientific literature. Theoretically, it reflects material's ability to recover to its original state in an infinite time after subjection to constant shear forces, at the constant temperature [2]. Practically, however, thixotropy is measured within a finite time period. A widely accepted definition, consistent with the IUPAC terminology and used in this study, is: the continuous decrease of viscosity with time when flow is applied to a sample that has been previously at rest and the subsequent recovery of viscosity in time when the flow is discontinued [9]. Two rheological tests were compared: a stan-

Phase	INCI name	Concentration %w/w
A	Glyceryl stearate, cetareth-20, cetareth-12, cetearyl alcohol and cetyl palmitate	4.0
	Butyrospermum parkii	4.0
	Ethylhexyl palmitate	20.0
	Propylparaben	0.2
B	Glycerol	3.0
	Methylparaben	0.2
	Carbomer	0.1–0.5
	Xanthan gum	1.5–3.5
	Triethanolamine	q.s. to pH 6.5
	Aqua	up to 100.0

Tab. 1 The formulation of the semisolid w/o emulsion used in the study

standard test used to measure the level of thixotropy, the hysteresis loop test, and a less known, the three-step thixotropy test. Texture analysis offers a range of methods for the evaluation of semisolid systems. The immersion/de-immersion (penetration/withdrawal) test using a cylindrical probe is one of the most recognised due to its versatility [10]. As shown in a recent study [11], the results obtained from the penetration test can also be used to identify the yield value and to measure the level of thixotropy. It was of interest to assess how this innovative method relates to rheometer-based methods. Overall, the purpose of this study was to explore and compare a range of rheological and texture analysis methods, both commonly used and alternative, employed in the quantification of the yield value and level of thixotropy of cosmetic semisolids.

Materials and Methods

A simple o/w emulsion (Tab. 1) was used as a model formulation. Two series of samples, containing a different rheological modifier, were used: with neutralised carbomer (0.1 %, 0.2 %, 0.3 %, 0.4 % and 0.5 % w/w) and with xanthan gum (1.5 %, 2.0 %, 2.5 %, 3.0 % and 3.5 %w/w). The instruments used in this study were: the air-bearing controlled-stress rheometer (RheoStress RS75, Haake, Germany), the T-bar viscometer (Brookfield DV-E, Brookfield, UK) and the texture analyser (TA.XT Plus, Stable Micro Systems, UK). All measurements were carried out in triplicate, at the temperature of 22 °C. Five different methods were used to assess the yield stress. The simplest was a low-rotation T-bar test on a viscometer (at 5 rpm with helipath, using a probe T 92). Readings were taken after the first 20 seconds, then at 10-second intervals until 60 seconds, calculating the average of the 5 values. The air-bearing rhe-

ometer was used for the shear stress sweep (0.5-500 Pa), the shear rate sweep (300-0.01 s⁻¹), and the oscillatory stress sweep (0.5-500 Pa at 1 Hz). For all rheometer measurements, the 35 mm serrated parallel plate was used, with the gap of 0.5 mm. The texture analyser was employed to perform the immersion/de-immersion test (with a one inch-diameter cylindrical probe made of perspex). The pre-test speed was 1mm/s, both test and post-test speed 2 mm/s, the immersion distance 8 mm and the trigger force 1 g. The point at which the material yields was shown as the inflection point on the positive (immersion) curve. The level of thixotropy was assessed using three tests, two on the rheometer and one on the texture analyser. The standard hysteresis

loop test was run from 0.01 to 300 s⁻¹ and back to 0.01 s⁻¹, each step taking 60 seconds. The three-step thixotropy test was performed using a succession of low, high and low shear rates, in this case 10, 250 and 10 s⁻¹, respectively, each step taking 30 seconds. The texture analyser was used to perform the repeated immersion/de-immersion test (3 repetitions), using the same settings as for the yield stress test. Statistical analysis was performed in the form of simple linear regression in the cases where one variable was experimentally changed (polymer concentration) and the other variable measured as dependant (rheological parameters). When comparing the results obtained from the two methods, a correlation analysis was performed, followed by the F-test of overall significance where appropriate. The significance threshold was 0.05.

Results and Discussion

Quantifying the Yield Stress

The results were analysed for their consistency with theoretical predictions, as well as statistically for correlations between the methods and/or the concentration ranges. Fig. 1 shows the results of yield stress obtained using a low-rotation T bar

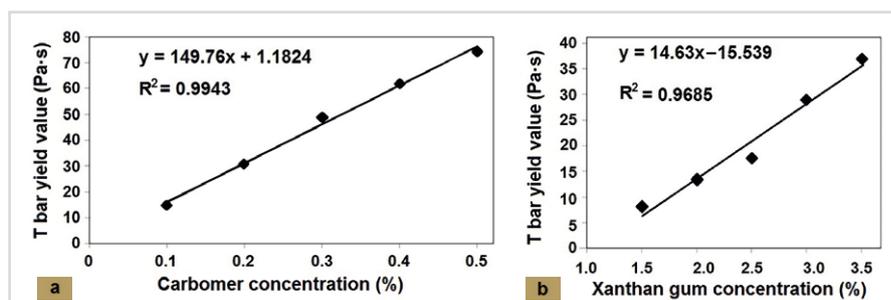


Fig. 1 Linear regression fit between increasing polymer concentration and yield value, measured using a low-rotation T bar test; (a) emulsions with carbomer; (b) emulsions with xanthan gum

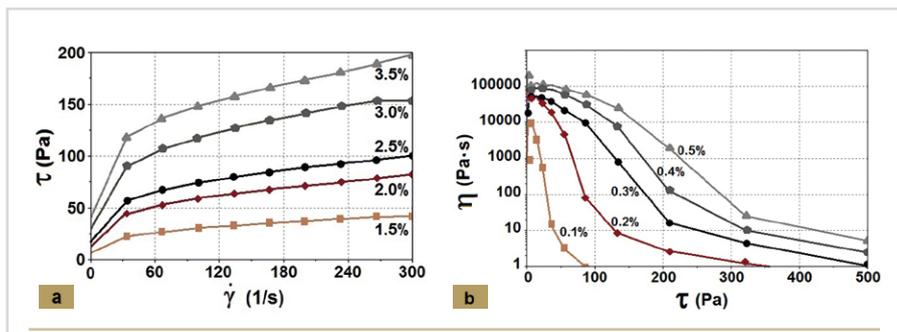


Fig. 2 Shear rate sweep results for the xanthan gum emulsions (a) and shear stress sweep results for the carbomer emulsions (b)

test plotted against increasing polymer concentration. Although this test officially reports viscosity values, there is an argument that the “T bar viscosity” is predominantly a measure of yield stress/gel strength, rather than viscosity. A true viscosity measurement requires the sample to be sheared between two close-fitting, geometrically-defined surfaces, such as the case with the cone-and-plate or parallel plate rheometer [2], but not the T bar. Therefore, the rheological response obtained at very low shear rates (in this case, related to the rotational speed of 5 rpm) could be considered a reflection of their yield stress. **Fig. 1** reveals a very good linear regression fit between increasing polymer concentration and the T-bar yield values obtained for both carbomer ($R^2=0.9943$) and xanthan gum-based emulsions ($R^2=0.9685$), hence corresponding with the theoretical predictions.

Typical results obtained from continuous flow rheology measurements, shear rate sweep and shear stress sweep, for the xanthan gum and carbomer emulsions are shown in **Fig. 2a** and **Fig. 2b**, respectively.

The standard shear rate sweep was carried out from high to low values (in this case from 300 to 0.01 s^{-1}) in order to mi-

nimise experimental artefacts, e.g. the fracturing of some samples and possible “noise” at low shear rates. The value of 0.01 s^{-1} was the lowest shear rate available. All ten model emulsions were clearly shear-thinning materials of plastic type (i.e. with yield stress). The results are presented in the form of flow curves (shear stress vs. shear rate), whereby the yield stress is detected as an intercept on the Y axis (**Fig. 2a**). There is a number of mathematical models used to describe this type of

non-Newtonian flow [12]. Our previous work [13] has shown that the Hershel-Buckley model was the best fit for the semi-solid emulsion systems, hence it was used in this study. In the Hershel-Buckley equation shown below

$$\tau = \tau_0 + k \cdot \dot{\gamma}^n$$

the following denotations apply:

- τ – Shear stress
- $\dot{\gamma}$ – Shear rate
- τ_0 – Yield stress
- k – Consistency index
- n – Flow index

The calculated values of τ_0 from the respective Hershel-Buckley equations were presented and analysed alongside the measured values obtained at the lowest shear rate (0.01 s^{-1}) for the ten samples (**Tab. 2**). Unlike the T bar values, neither of these two methods have shown the expected linear relationship between the yield stress results and increasing polymer concentrations.

In the case of shear stress sweep, theoretically, the viscosity curve shows an inflection at the point where material yields.

Polymer (% w/w)	Yield stress method					
	T bar with helipath (Pa·s)	Shear rate sweep (Pa)	Shear rate sweep modelled (Pa)	Shear stress sweep (Pa)	Oscillatory stress sweep (Pa)	Immersion/de-immersion (g)
Carbomer						
0.1 %	14.83	31.05	30.10	35.82	7.90	2.7
0.2 %	30.88	43.39	41.62	55.62	11.19	3.2
0.3 %	48.70	42.22	46.3	86.30	15.81	3.5
0.4 %	61.96	20.74	18.52	207.70	18.79	4.0
0.5 %	74.17	11.82	-16.45	207.70	22.33	5.7
Xanthan gum						
1.5 %	8.04	7.07	3.79	9.60	1.18	2.0
2.0 %	13.55	13.95	7.42	207.70	1.67	2.2
2.5 %	17.46	17.88	7.74	35.85	2.36	3.8
3.0 %	28.94	31.22	14.77	55.62	2.81	4.9
3.5 %	36.92	41.75	18.93	55.62	7.92	5.8

Tab. 2 Yield stress values obtained using six different methods, five experimental and one based on mathematical model (Hershel-Buckley equation)

In practice, the curve presents a yielding region, rather than a point (Fig. 2b), which has made the detection of the yield stress difficult. In addition to that, only higher concentration samples have shown measurable viscosity values within the whole region of the stress sweep, from 0.5 to 500 Pa (Fig. 2b). For the purpose of this study, the yield point was detected as the stress at which viscosity is reduced by the factor of 10 from its initial, zero-shear viscosity. This stress could be interpreted as the point where the sample has achieved its 90 % yield. The results obtained by the shear stress sweep method, presented in Tab. 2, show an inconsistent trend regarding increasing polymer concentration for both carbomer and xanthan gum. Consequently, neither shear rate sweep nor shear stress sweep appear to be a reliable formulator's tool for the testing of yield stress. Unlike the continuous flow tests discussed above, the oscillatory (dynamic) rheological tests involve the application of a sinusoidally oscillating (clockwise/counter-clockwise) shear stress to a sample and the subsequent measurement of the shear

strain [2]. There are two main independent variables that could be used, shear stress and the frequency of oscillation, resulting in the oscillatory stress sweep and frequency sweep method, respectively. A distinct advantage of dynamic tests is that they could be performed at very low shear stresses, normally below yield point, allowing an insight into the internal structure of a semisolid without destroying it [14]. They measure both elastic and viscous rheological properties of the material (e.g. elastic modulus G' and viscous modulus G''), and could provide a range of complex parameters that quantify

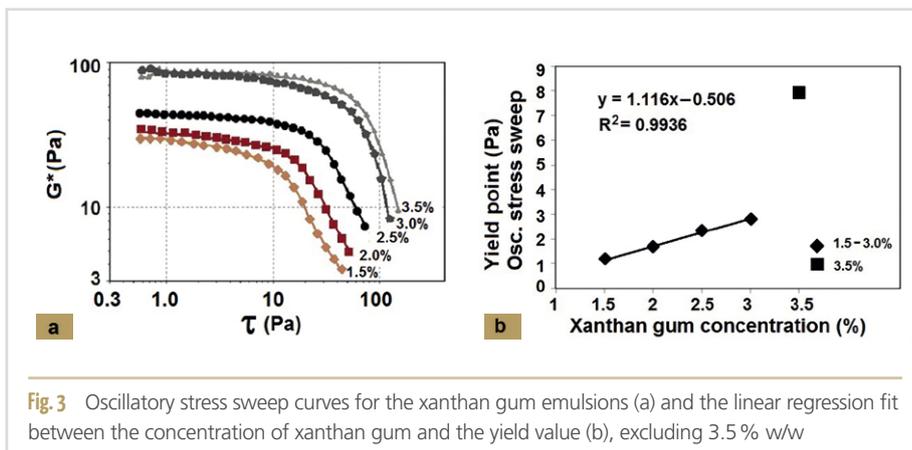


Fig. 3 Oscillatory stress sweep curves for the xanthan gum emulsions (a) and the linear regression fit between the concentration of xanthan gum and the yield value (b), excluding 3.5 % w/w

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the viscoelastic balance within the sample (e.g. phase angle δ and complex modulus G^*).

This study has employed an oscillatory stress sweep from 0.5 to 500 Pa, at the constant frequency of 1 Hz. **Fig. 3a** shows the changes in the complex modulus G^* with increasing oscillatory stress, in the case of xanthan gum emulsions. Complex modulus, mathematically defined as the ratio of stress to strain, reflects the resistance to deformation of the sample, i.e. its rigidity. Complex modulus stays constant under low shear stresses (known as viscoelastic region), but it decreases once the stress reaches the point of breaking the original structure (yield stress).

Similarly to the continuous stress sweep method described above, in the oscillatory stress sweep the yield stress is detected as an inflection point on the G^* curve (**Fig. 3a**). Since it is again a region, not a point, the reported value depends on the method chosen. For example, from the same oscillatory stress sweep run, the yield value could be determined (arbitrarily) as the first downward inflection on the complex modulus G^* curve, which coincides with the upward inflection of the phase angle curve (not shown). It could also be determined as the stress at which the phase angle reaches 45° , the rationale being that this is the point of transition from a more solid (elastic-dominant) to a more liquid (viscous-dominant) behaviour. Yet another way, used in this study, is to take the stress value at which the sample has softened 10 times, i.e. G^* has decreased by the factor of 10 (**Tab. 2, Fig. 3a**).

It is interesting to note that the yield values of the first 4 samples form an almost ideally linear relationship ($R^2 = 0.9936$) with the concentration of xanthan gum (**Fig. 3b**), while the most concentrated sample performed differently. This phenomenon, which was not detected using less sensitive methods (e.g. **Fig. 1b**) is probably due to the level of polymer entanglement and the resulting secondary chemical bonds at that concentration, which should be further explored. In the case of carbomer emulsions, the correlation was very good for the whole range ($R^2 = 0.9960$).

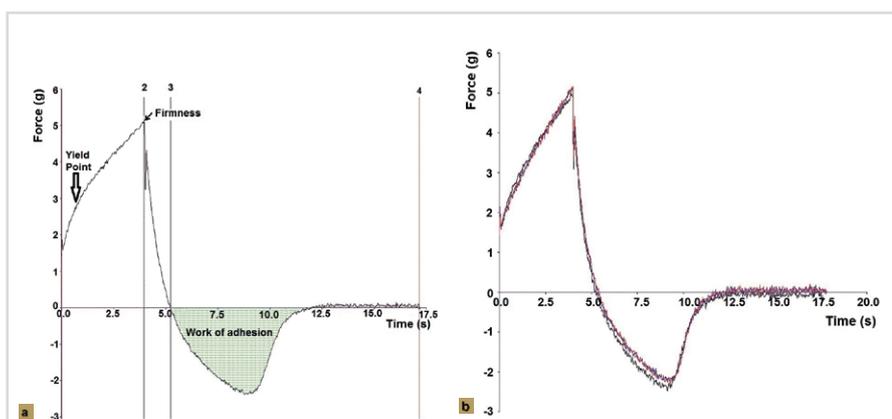


Fig. 4 Typical curves obtained using the immersion/de-immersion test on the texture analyser for the detection of yield value (a) and the repeated immersion/de-immersion (deformation) test for the detection of the level of thixotropy (b)

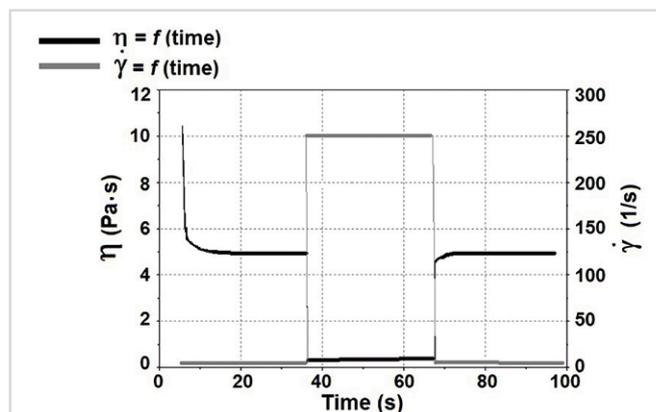


Fig. 5 Three-step thixotropy response of the 2.5 % xanthan gum sample (viscosity curve shown as black line; shear rate curve shown as gray line)

The texture analyser has been commonly used for the evaluation of textural properties of semisolid cosmetic products, for example firmness, consistency, cohesiveness, stringiness and spreadability [10]. However, a recent paper [11] has demonstrated a wider potential of this instrument, including the yield stress and thixotropy tests. Using the suggested yield stress method, we have tested the two series of emulsions, with very consistent results. **Fig. 4a** shows a typical immersion/de-immersion curve, with the inflection on the ascending part indicating the yield value. The yield stress data obtained from the immersion/de-immersion test, alongside the data obtained by other methods used in this study, are presented in **Tab. 2**. There was again a strong linear regression fit between the polymer concentration and the yield stress detected using the texture analyser (e.g. $R^2=0.9652$ for xanthan gum emulsions).

Overall, the analysis of the results from **Tab. 2** has shown that each of the instruments used in this study could be successfully employed for the measurement of the yield stress. However, commonly used shear rate sweep and shear stress sweep have not provided consistent results and should be replaced with the oscillatory stress sweep.

Quantifying the Level of Thixotropy

A standard test used to measure the level of thixotropy is two-directional shear rate sweep that produces two non-overlapping curves (up and down curve). The area between the curves is known as the hysteresis area and is widely considered as the measure of thixotropy [2]. In this study, the hysteresis loop test was compared with a different approach, a three-step thixotropy test. The disadvantage of the standard hysteresis loop test is that the two variables (shear rate and time) act

simultaneously, which means that their effect on the sample cannot be distinguished. This disadvantage could be overcome by using the method of stepwise changes in shear rate or shear stress [9]. The three-step thixotropy test is a version of this method, whereby the shear rate changes from low, to high and back to low in three separate steps. Since each step involves constant shear rate, the method allows the effect of time to be observed separately from shear. The three-step thixotropy test used in this study measured the percentage (%) recovery of the sample subjected to the low shear (10 s^{-1}), very high shear (250 s^{-1}) and again low shear (10 s^{-1}) for 30 seconds each time.

A typical response with high recovery rate is shown in Fig. 5. The level of thixotropy is calculated by taking the viscosity value from the end of the first step (as average of 10 values), the same from the end of the third step and calculating the % difference. The result could be expressed as the % recovery or the % loss. This test, similarly to the hysteresis loop test, measures the level of instantaneous response by the sample, not the time necessary for the recovery to

take place, for which a different type of test should be employed.

The results of the hysteresis loop test for the xanthan gum emulsions are shown in Fig. 6a.

A strong linear relationship between the level of thixotropy measured by the three-step method (expressed as % loss) and the increasing concentrations of polymer was observed (e.g. $R^2=0.9807$ for the carbomer emulsions). Interestingly, the comparison of the results obtained by the three-step and the hysteresis loop methods (Fig. 6b) has revealed a good, although not linear correlation.

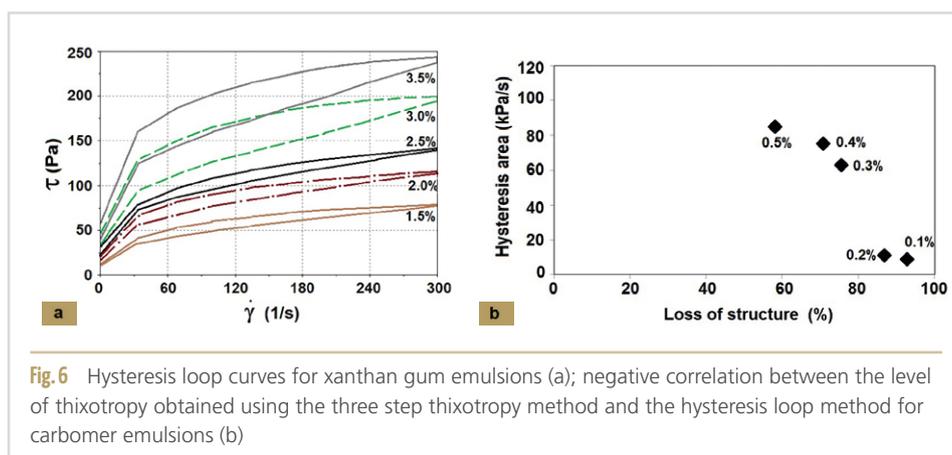


Fig. 6 Hysteresis loop curves for xanthan gum emulsions (a); negative correlation between the level of thixotropy obtained using the three step thixotropy method and the hysteresis loop method for carbomer emulsions (b)

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The thixotropy test was also performed on the texture analyser by repeated immersion/de-immersion, as suggested by Tai et al [11]. **Fig. 4b** represents a typical response of the repeated deformation test, where three measurements have been performed in short succession on the same area in an attempt to measure the level of thixotropy. The work of adhesion, i.e. the area under the negative curve (**Fig. 4b**) was selected as a relevant parameter. All tested samples have shown the same pattern – the largest difference in all measured parameters was seen between the first and the second measurement, while subsequent measurements have shown almost no difference. Consequently, the test was limited to three repetitions. The work of adhesion of the second immersion was used as the parameter reflecting the level of thixotropy in the comparative analysis of the three methods. The repeated immersion/de-immersion test has displayed a positive correlation with the hysteresis loop and the three-step method, although not statistically significant.

Conclusion

The most consistent yield stress results were obtained using the low-rotation T-bar test, followed by the oscillatory stress sweep test and the novel immersion/de-immersion texture analyser test. Providing all three instruments are available, a formulator is advised to choose the oscillatory stress sweep as the most theoretically robust, informative and reproducible. However, there must be a well-established criterion for the determination of the yield stress from the yield region, e.g. the stress at which the initial viscosity has been reduced 10 times.

In terms of the finite-time thixotropy measurements, both hysteresis loop test and three-step thixotropy test have proven consistent in assessing immediate effects of the application of shear to the semisolid emulsion structure. If only texture analyser was available, however, the repeated immersion/de-immersion test could also be used to give a good indication of the level of thixotropy in semisolid emulsions.

By changing the conditions of each test, it is possible to produce an infinite number of variations; the merits of each should be established with regard to the purpose of the test (e.g. physico-chemical characterisation, stability prediction, production specification or sensory characterisation).

Note

Shorter version presented at the 29th IFSCC Congress, Orlando, USA, 31st Oct – 2nd Nov 2016

Acknowledgements

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