A comparison of rheological and texture analysis methods for the assessment of viscoelasticity

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Introduction

It is of practical, as well as theoretical interest, to determine the position of a particular semisolid product on the viscoelastic scale.

Dynamic (oscillatory) rheology is a standard method to measure viscoelasticity, applying the oscillating shear stress and measuring the resulting strain [1]. There are two principal approaches, ***stress sweep*** and ***frequency sweep,*** and four commonly used parameters: ***elastic modulus G',*** ***viscous modulus G",*** ***phase angle δ*** and ***complex modulus G*.***

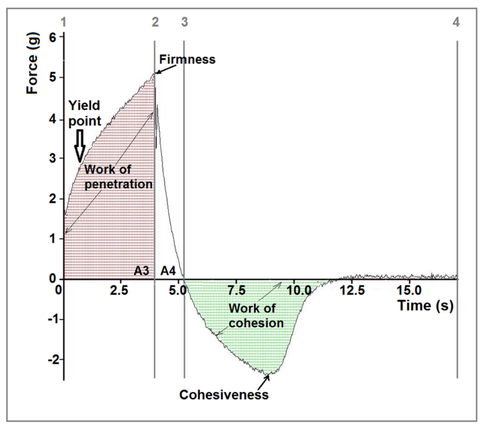


Fig. 1 Typical curve obtained by texture analyser, expressed as force vs. time

For the assessment of semisolids, **texture analysis** employs the **immersion/de-immersion (penetration) test** using a cylindrical probe. A typical response (Fig. 1) consists of the positive and negative curves, with four established parameters: ***firmness, consistency*** (work of penetration), ***cohesiveness and work of cohesion***. It can also measure the ***yield value*** and ***the level of thixotropy*** [2,3].

This study explores the two novel TA parameters: ***stringiness*** (the distance at which the sample ruptures when the probe rises over the surface) and ***resilience*** (the ratio between areas A3 and A4, Fig. 1), and compares them with oscillatory parameters.

Materials and Methods

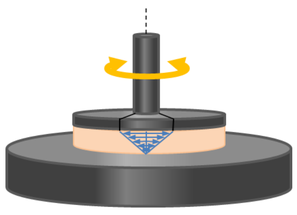
A simple o/w emulsion (Table I) was used as a model formulation. Two series of samples, each containing a different rheological modifier, were used: with neutralised **carbomer** (0.1%, 0.2%, 0.3%, 0.4% and 0.5% w/w) and with **xanthan gum** (1.5%, 2.0%, 2.5%, 3.0% and 3.5%w/w).

Table I The formulation of the model semisolid emulsion

Phase	INCI name	Concentration %w/w
A	Glyceryl Stearate, Ceteareth-20, Ceteareth-12, Cetearyl Alcohol and Cetyl Palmitate	4.0
	Butyrospermum Parkii	4.0
	Ethyl Hexyl Palmitate	20.0
	Proplyparaben	0.2
B	Glycerol	3.0
	Methylparaben	0.2
	Carbomer	0.1- 0.5
	Xanthan Gum	1.5 - 3.5
	Triethanolamine	up to pH 6.0
	Aqua	up to 100.0

The instruments used in this study were an air-bearing controlled-stress rheometer (RheoStress RS75, Haake, Germany) and a texture analyser (TA.XT Plus, Stable Micro Systems, UK). Rheological measurements were carried out using a 35-mm serrated parallel plate, with the gap of 0.5 mm. The methods used were stress sweep (0.5-500 Pa at 1 Hz) and frequency sweep (0.01-10 Hz at 10 Pa).

a)



b)

Fig. 2 Visual representation of the oscillatory rheology method (a) and texture analysis method (b)

The texture analysis method used in this study was 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 2mm/s, the immersion distance 8 mm and the trigger force 1g. All measurements were carried out in triplicate, at a constant temperature of 22^0C.

Results and Discussion

Since most oscillatory rheology is performed below the yield value, the first step was to detect the limits of the 'viscoelastic range' for each sample, i.e. the stress levels at which its internal structure stays intact. ***Stress sweep tests*** form 0.5 to 500 Pa have revealed that the structure breakdown in most samples happened at above 20 Pa (e.g.. Fig. 1), hence it was decided that all ***frequency sweep tests*** would be carried out at 10 Pa.

Fig. 4 presents the elastic modulus results obtained for the two sets of samples, showing the expected increase in elasticity with polymer concentration and the frequency of oscillation [4].

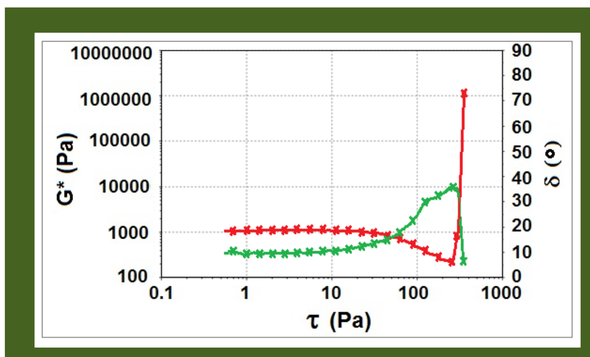


Fig.3 Oscillatory stress sweep results for 0.1% carbomer, showing a decrease in the **complex modulus (G*,** red) and an increase in the **phase angle (δ,** green**)** at stress levels above 20 Pa (yield region)

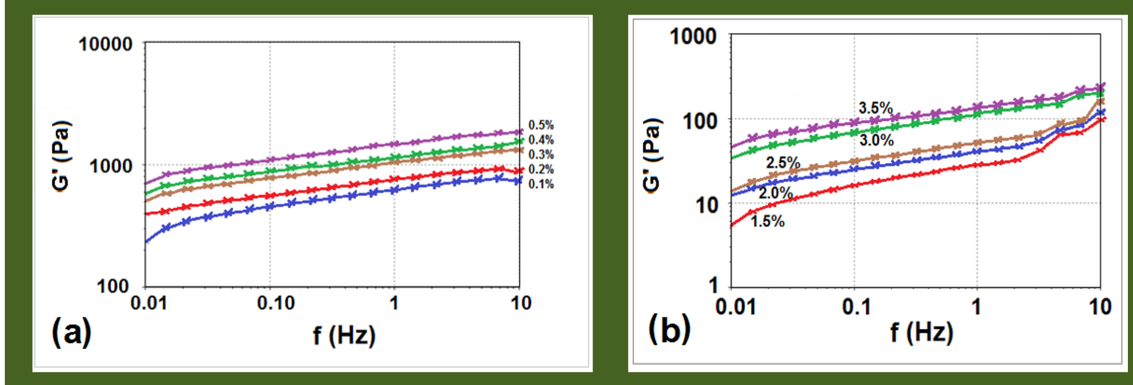


Fig. 4 Elastic modulus (G') for the emulsion samples with increasing carbomer (a) and xanthan gum concentrations (b)

Table II Texture analysis parameters of the emulsion samples stabilised with carbomer and xanthan gum

Polymer (% w/w)	TA parameters of the test emulsions					
	Firmness (g)	Consistency (g.s)	Cohesiveness (g)	Work of Cohesion (g.s)	Resilience (A4/A3)	Stringiness (mm)
Carbomer						
0.1%	5.13	17.17	-2.39	-8.33	0.16	7.25
0.2%	8.67	25.53	-4.28	-15.04	0.18	6.50
0.3%	13.57	40.14	-6.78	-21.07	0.19	6.00
0.4%	18.06	54.66	-8.80	-25.27	0.19	5.75
0.5%	22.56	67.43	-10.20	-28.67	0.21	5.50
Xanthan gum						
1.5%	3.45	12.61	-1.45	-4.69	0.18	8.20
2.0%	4.93	17.48	-2.22	-7.80	0.19	8.00
2.5%	5.56	19.64	-2.60	-8.98	0.21	7.50
3.0%	7.29	24.30	-3.34	-10.98	0.20	7.00
3.5%	9.06	29.86	-3.47	-11.92	0.22	6.00

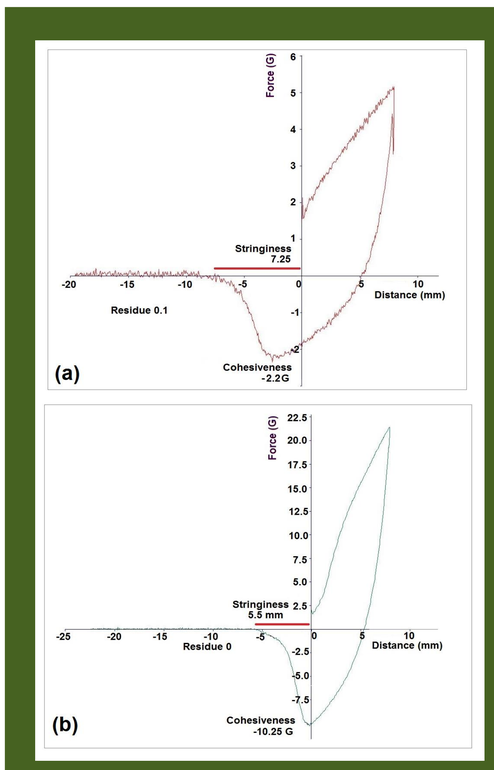


Fig. 5 Typical curve obtained by immersion/de-immersion method on TA, expressed as force vs. distance. **Stringiness** is defined as the distance that the product is extended during de-immersion stage before separating from the probe; a) emulsion with 0.1% carbomer; b) emulsion with 0.5% carbomer

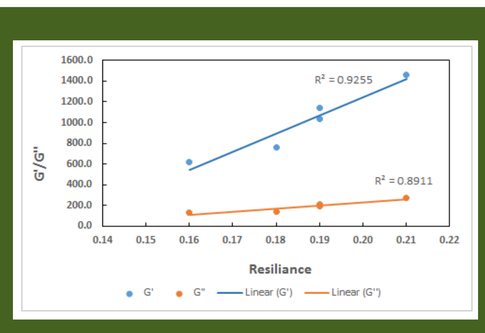


Fig. 6 Linear regression fit between a TA parameter resilience and two oscillatory parameters, **elastic modulus G'** and **viscous modulus G''**, for the series of emulsions with carbomer

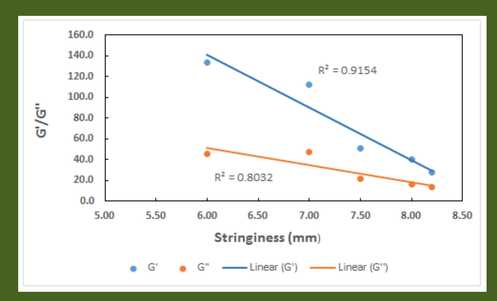


Fig.7 Linear regression fit between a TA parameter stringiness and two oscillatory parameters, **elastic modulus G'** and **viscous modulus G''**, for the series of emulsions with xanthan gum

Conclusion

This study has found that a novel TA parameters ***resilience*** and ***stringiness*** correlate with the key oscillatory parameters, the ***elastic (G')*** and ***viscous (G") modulus***. Resilience mildly increases with the increase in polymer concentration for both carbomer and xanthan gum–stabilised emulsions, alongside G' and G''. Stringiness has shown the opposite trend, i.e. negative correlation with both G' and G''.

In conclusion, if carefully interpreted, TA parameters resilience and stringiness could be used to assess the level of viscoelasticity of cosmetic semisolids.

References:

[1] Brummer, R., *Rheology essentials for cosmetic and food emulsions*, Springer, Hamburg (2006); [2] Tai, A., Blanchini, R. and Jachowicz, J. Texture analysis of cosmetic/ pharmaceutical raw materials and formulations, *Int. J. Cosmet. Sci*. **36**, 291-304 (2014); [3] Tamburic, S, Sisson, H, Cunningham, N and Stevic, M.C. Rheological and texture analysis methods for quantifying yield value and level of thixotropy, *SOFW Journal*, **143** (6), 24-30 (2017); [4] Craig, D.Q., Tamburic, S., Buckton, G. and Newton, J.M. An investigation into the structure and properties of Carbomer 934 gels using dielectric spectroscopy and oscillatory rheometry. *J. Control. Rel.*, **30**, 213-223 (1994)