COMBINING INSTRUMENTAL AND SENSORY EVALUATION TO ASSESS APPLICATION CHARACTERISTICS OF SKINCARE EMULSIONS

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Introduction

Sensory (application) properties of skincare products are crucial for their consumer acceptance. They are dependent, often in a complex way, on the multitude of formulation variables, including the type and concentration of emulsifiers, thickeners, emollients and humectants (1). There have been many successful attempts to relate the physicochemical properties of various raw material classes to their sensory properties (e.g. 2). However, when many ingredients are combined in the form of skincare products, these relationships become less reliable.

In this study, two types of instrumental methods were used to characterise two series of semisolid skincare emulsions based on different emulsifier systems, and combined with a trained sensory panel. Data analysis has explored correlations that may be used in practical formulation work.

Aim

The aim of the study was to investigate the effect of formulation variables on the rheological, textural and sensory properties of skincare emulsions. In addition, it was of interest to explore possible relationships between instrumental and sensory parameters.

Materials

A skincare emulsion with a novel antioxidant active, Cocoa extract, was chosen for this study. In line with the 'green' formulation trend, no ethoxylated emulsifiers were used. The first series was based on a non-ionic emulsifier system, **sorbitan stearate & sucrose cocoate**, while the second series relied on the hydrophobically modified polyacrylic acid polymer, **acrylates C10-30 alkyl acrylate crosspolymer** (***Tables 1 & 2***). The concentration of thickeners, as well as the type and concentration of various emollients, were varied in a systematic manner within the two series. The pH of all formulations was adjusted to be between 5.2 and 5.5. A commercial preservative mix at 0.5% was used in all cases.

Table I. Non-ionic cream formulations

	Ingredients	F1	F2	F3	F4	F5	F6	F7	F8
Oil phase	Sorbitan Stearate &Sucrose Cocoate	5.5	8.25	5.5	5.5	8.25	5.5	5.5	8.25
	Isopropyl Myristate	9.5	9.5	14.25	9.5	9.5	9.5	4.5	4.5
	Dicaprylyl Carbonate	4.5	4.5	4.5	6.75	6.75	4.5	9.5	9.5
Water phase	Aqua	69.05	66.3	64.3	66.8	64.05	68.95	68.95	66.3
	Carbomer	0.20	0.20	0.20	0.20	0.20	0.30	0.30	0.20
	Cocoa Extract	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
	Propylene Glycol	10	10	10	10	10	10	10	10
	Triethanolamine (10% solution)	quantum satis							

Table II. Polymeric cream formulations

	Ingredients	F1	F2	F3	F4	F5	F6	F7	F8	F9
Oil phase	Alkylacrylate crosspolymer	025	0.25	0.25	0.37	0.25	0.25	0.25	0.25	0.37
	Carbomer	0.20	0.20	0.30	0.30	0.20	0.20	0.20	0.20	0.20
	Paraffinum Liquidum	10	10	10	10	10	10		10	10
	Ethylhexyl Stearate	8	8	8	8	-	8	8	-	-
	Caprylic/capric Triglyceride	4.5	4.5	4.5	4.5	4.5	-	4.5	4.5	4.5
	Oleyl Oleate					8	4.5	10	16	16
Water phase	Aqua	65.8	64.68	64.7	64.8	64.8	64.8	64.8	56.8	56.58
	Propylene Glycol	10	10	10	10	10	10	10	10	10
	Cocoa Extract	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
	Triethanolamine (10% solution)	quantum satis								

Figure 1. RheoStress instrument used in rheological testing

Methods

Rheological parameters were obtained using a cone-and-plate viscometer **RheoStress** (Haake, Germany). Continuous flow tests were performed by increasing a shear stress from 0 to 100 Pa and decreasing it back to 0, each stage taking 1 minute. Textural properties were assessed by **Texture Analyser TA.XT plus** (Stable Micro Systems, UK). The pre-test speed of a probe was set up at 1.0 mm/s, the test speed (penetration and withdrawal) at 1.5 mm/s and the probe depth at 15 mm. The probe used was a plastic cylinder with a diameter of 2 cm. Both tests were performed at room temperature (21 C), with at least three repeats for each sample.

Figure 2. Loading of a sample on Texture Analyser

Sensory parameters were obtained from the panel of 12 assessors, trained in the use of reference samples in conjunction with the test terminology, as recommended in the literature (3). The parameters tested were: ***texture, initial pick up, skin feel on application, after feel, gloss, wetness, spreadability, amount of residue and absorbency***; the scale used was 0-10 (very low to very high level of each attribute).

Results and Discussion

The results of the two instrumental tests (average values) from the two series of selected samples are presented in ***Table III***. Yield value (yield stress), viscosity and hysteresis area were obtained from the rheological measurements, while firmness and work of penetration were textural parameters from Texture Analyser. **Yield value**, as a measure of the elastic component of the plastic flow behaviour, was generally higher in the group based on the polyacrylic acid stabilising system, which was not surprising given the elastic network formed in these samples. **Hysteresis area** is known to be the measure of thixotropy, a time-dependent rheological behaviour, reflecting reversible breakdown of the system under the influence of external force (4). Interestingly, the hysteresis area has shown a relatively good level of correlation with firmness ($R^2 = 0.72$).

Table III. Rheological and textural parameters for the test samples

	Yield value (mPa)	Viscosity at $100s^{-1}$ (mPa.s)	Hysteresis area ($Pa.s^{-1}$)	Firmness (g)	Work of penetration (g.sec)
non-ionic					
F1	454	1,565	708	13.281	91.512
F2	445	2,770	5152	32.58	246.095
F5	604	1,090	7506	27.68	183.985
F6	529	1,927	3112	28.146	201.337
F8	495	1,918	4883	18.144	138.633
polymeric					
F1	579	1,059	114	11.048	78.979
F3	620	1,158	301	11.811	82.137
F4	454	1,011	342	11.141	80.935
F7	687	941	369	11.998	81.611
F9	537	1,806	238	12.045	86.522

Figure 3 shows a series of penetration and withdrawal curves for the selected non-ionic emulsion samples. **Firmness** is defined as the highest point on the penetration curve, while **work of penetration** is the area under the curve (5), both reflecting the resistance showed by the sample to the penetrating force of the cylinder probe.

It is clear from ***Table 3*** that viscosity correlates well with both firmness and work of penetration. In the case of non-ionic samples, the highest values for all three parameters belong to sample F2, followed by F6; the same applies to F9 and F3 in the polymeric group. Direct relationship between rheological and textural parameters has already been reported (6), while investigating a series of semisolid emulsions based on silicone emulsifier.

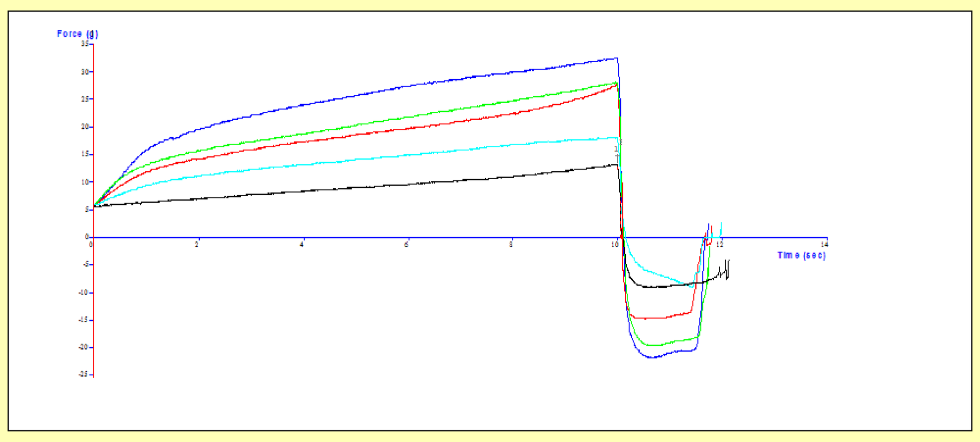

Figure 3. Textural analysis: penetration and withdrawal curves for non-ionic formulations

Four samples have been chosen as most successful sensory representatives from the two series: F2 and F6 from the non-ionic group and F3 and F9 from the polymeric group. Their sensory characteristics are presented in the form of 'spider' diagram in ***Figure 4***. The overall 'winner' was a non-ionic sample F6, which was given the highest score for the texture, skin feel on application, after feel and absorbency, with a low score of the amount of residue and solid scores for initial pick up and spreadability.

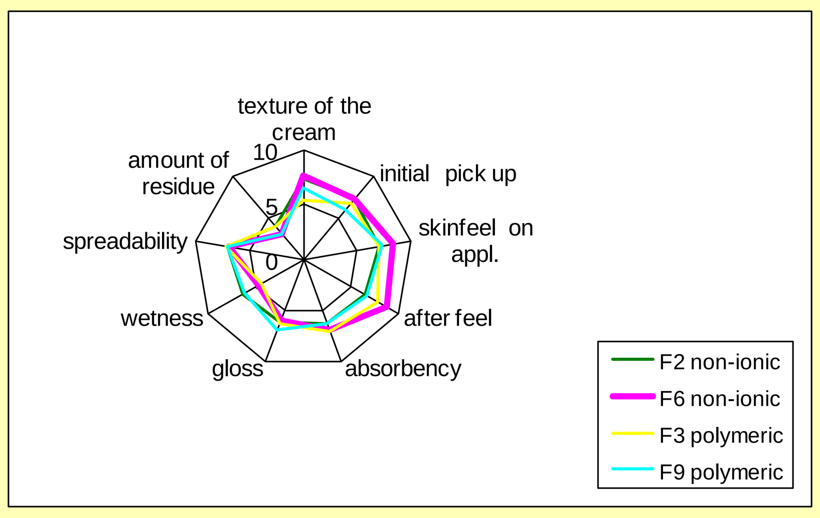

Figure 4. Sensory analysis: panel data for the four most successful samples

When analysing both series of samples together, the correlation analysis has revealed only two relationships with R^2 higher than 0.70 - between viscosity and the texture of the cream and viscosity and initial pick up. However, when looked separately, polymeric creams in general have shown much better correlation between instrumental and sensory data. For example, the correlation coefficient between viscosity and texture was 0.9775 (in the positive direction), while the same set of samples have shown high negative correlation with the amount of residue, with $R^2 = 0.9040$.

Conclusions

- Direct relationship between continuous flow rheology and texture analysis was obtained in both series of samples.
- Viscosity has shown the highest potential to predict sensory responses, especially cream texture and initial pick up.
- Polymer-based series has shown significantly higher correlation between selected instrumental and sensory parameters (e.g. viscosity/texture and viscosity/the amount of residue), compared to the non-ionic series, therefore

some emulsion structures tend to behave more predictably in terms of sensory properties than others.

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