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Study of sensory properties of emollients used in cosmetics and their correlation with physicochemical properties

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Synopsis

Eight liquid emollients (mineral oil, sunflower oil, squalane, decyl oleate, isopropyl myristate, octyldodecanol, dimethicone, and cyclomethicone) were characterized by instrumental and sensory methods and evaluated to determine the relationship between sensory and instrumental measures. Sensory analysis was carried out by a panel of 14 assessors, who evaluated the following attributes: difficulty of spreading, gloss, residue, stickiness, slipperiness, softness, and oiliness. The physicochemical properties measured were spreadability (at one-half minute and at one minute), viscosity, and superficial tension.

Data collected were statistically analyzed by analysis of variance (ANOVA), principal component analysis (PCA), and linear partial least squares regression analysis (PLS). In consideration of their physicochemical characteristics, the studied emollients were sorted into three groups, in which the silicones distinctly separate from the rest. Sensory characteristics enabled the discrimination of four groups of emollients where, besides the two silicones, isopropyl myristate was also differentiated. PLS revealed that emollient sensory attributes could be well predicted by instrumental measurements.

INTRODUCTION

The inclusion of emollients in cosmetic emulsions is a common practice, independent of the final use of the emulsion. In consideration of their action on the skin, emollients can be regarded as a replacement of natural lipids, as they contribute to water retention by the stratum corneum. They result in a smoother, more elastic, and lubricated skin, and impart a pleasant and comfortable skinfeel (1–3).

Lipophyllic emollients offer a wide variety of sensations when applied to the skin (waxy,

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greasy, oily, dry, astringent, sticky, velvety, etc.) and may have great incidence on the sensory characteristics of the cosmetic emulsions in which they are included (4–6).

Emollients also have major incidence on the physicochemical properties, such as consistency and spreadability, of the emulsions in which they are included. These characteristics are very important to achieve adequate efficacy and user acceptance of the products.

In general, there are few bibliographical references about sensory evaluation of cosmetics and, particularly, of emollients. In 1971 Goldember and De la Rosa (4) found that it was possible to quantify the skinfeel of emollient cosmetics incorporated in a standard base by using a skinfeel index (SFI) (ratio of the initial slip of a product (scale of 1 to 5) to its total end feel (scale of 4 to 20).

Perceived skinfeel attributes of creams and lotions (appearance, rub-in, absorption, appearance of skin, immediate and delayed afterfeel) have been evaluated by Aust *et al.* (7) using a trained descriptive panel.

In 1991, Civille and Dus (8) used the skinfeel spectrum descriptive analysis (skinfeel SDA) method to characterize skin care products. They used strict protocols for manipulation and precisely defined terms to describe the qualitative properties and their relative intensities in each product. The evaluation process was divided into four categories: pick-up (firmness, stickiness, cohesiveness, peaking); rub-out (wetness, spreadability, thickness, absorbency); residual feel; and appearance, immediate and after 20 minutes (glossy, sticky, slippery, oily, waxy, greasy).

The aims of the present work were to: (a) characterize, from a sensory point of view, eight liquid emollients frequently used in cosmetic emulsions and (b) study the correlation between the sensory characteristics of the emollients and their physicochemical properties.

MATERIALS AND METHODS

SAMPLES

The following emollients were used: mineral oil (MO), sunflower oil (SO), squalane (SQ), decyl oleate (DO), isopropyl myristate (IPM), octyldodecanol (OD), dimethicone (DM), and cyclomethicone (CM). The selected emollients have different chemical structures, including silicones (linear and cyclical), hydrocarbons (saturated and unsaturated), esters, branched-chain fatty alcohols, and natural oils. Sunflower oil, a common oil in Uruguay, is a substance of cosmetic interest due to its high content of unsaturated fatty acids (9,10).

SENSORY ANALYSIS

Selection of attributes and training of judges. Initially the emollients were presented to a group of 14 sensory judges experienced in the evaluation of food, who lacked experience in cosmetic products. They were asked to describe the sensations experienced when emollients were spread on their skin and the sensations they perceived when they touched and observed the emollient film formed over the skin immediately after application. The descriptors to be employed in the evaluation of emollients were selected by open discussion, taking into account the previously mentioned terms and the ones cited in the bibliographical references (8,11).

Working conditions were normalized in order to train the panel of judges and obtain valid and reproducible results. This normalization included the definition of the selected terms, the establishment of the conditions of sensory evaluation, and the selection of references (anchors) to quantify the intensity of each attribute (Table I). Secondly, judges were trained to evaluate the attributes using structured 10-point scales of intensity, until consistent judgments from the panel were achieved (8,11).

Sample evaluation method. A balanced complete-block experimental design was carried out for duplicate evaluation of the samples during six sessions (three samples in each session). Structured ten-point scales anchored with "nil" and "high" were used to describe attribute intensity. The test was carried out in a sensory laboratory designed in accordance with ISO 8589 (12).

Before the test started a mixture of isopropyl alcohol and water (45/55) was used on the forearms of the judges in order to clean the zone, allowing it to dry. Three 4-cm-diameter circles were outlined on the internal side of the non-dominant forearm. The coordinator placed one drop of the sample in the center of one circle, and the judge rotated the sample with one finger in a circular manner 120 times within the circle, at a rate of two times per second and employing a metronome to control the rhythm. The evaluated attributes are shown in Table I.

INSTRUMENTAL ANALYSIS

The physicochemical properties selected were those that could be related to the capacity of emollients to spread and to the sensations they cause when applied to the skin. These physicochemical properties were spreadability, viscosity, and superficial tension.

Instrumental spreadability determination. Instrumental spreadability was determined by

Table I
Emollient Profile Descriptors

Evaluation instance	Attributes	Description	"Nil"	"High"
During application	Difficulty spreading	Difficulty in moving product over skin	Baby oil	Lanolin USP
After application	Gloss	Amount or degree of light reflected off skin	Corega®*	Baby oil
	Residue	Amount of product left on skin	Untreated skin	Hipoglos®**
	Stickiness	Force required to separate a finger from skin	Baby oil	Lanolin USP
	Slipperiness	Ease of moving two fingers over the skin	Lanolin USP	Baby oil
	Softness	Skin surface uniformity	Corega®	Glass
	Oiliness	Type of residue (non-wet liquid that leaves residue)	Untreated skin	Baby oil

* Stafford Miller Ireland, LTD.
** Lab. Andrómaco.

gravity action at one-half minute and at one minute, by discharging 500 μl of emollient at 20° ± 1°C over a glass dish. Two perpendicular diameters of the area occupied by the sample at a half minute and at one minute (S0.5 and S1) were measured and the mean calculated. Five determinations were performed for each emollient, and the homogeneity of variance of the data set was studied in order to verify that the values were comparable.

Viscosity determination. The viscosity of the emollients was measured employing a Brookfield LVT viscometer. Measurements were carried out employing Spindle No. 1 at 60 rpm. Samples were thermostatized at 20° ± 1°C. Ten determinations were performed for each emollient, and the homogeneity of variance of the data set was studied in order to verify if the values were comparable.

Surface tension determination. The surface tension of each emollient was determined by stalagmometry (13,14). The method consists of weighing a drop of emollient obtained from the end of a calibrated capilar tube and determining the surface tension by means of a calibration plot. In order to draw the calibration plot, ten drops of known surface tension standards [diethyl ether, ethyl alcohol (absolute), chloroform, and carbon disulfide (all HPLC grade), and distilled water] were accurately weighed in a milligram balance. The calibration plot that correlates drop weight with surface tension for each of the standards was obtained by regression analysis. A linear model with its y-intercept through the origin (direct proportionality), with a significance level of 95% (equation 1), was accepted:

$$P_{10} = 0.357 \times \sigma \qquad (1)$$

in which σ = surface tension (din/cm) and P_{10} = weight of 10 drops (mg).

Ten drops of each emollient were weighed and their corresponding surface tensions were determined by means of the calibration plot. All determinations were carried out at a constant temperature of 20° ± 1°C.

DATA ANALYSIS

A three-factor (assessor, sample, repetition) analysis of variance (ANOVA) was performed for all samples on the sensory data obtained. The mean rating and Fisher's least significant differences for each term were calculated by ANOVA. Principal component analysis (PCA) of mean ratings for each sensory and instrumental attribute was used to illustrate the relationship among variables and samples. All statistical analyses were performed using Statistica 5.1 software (StatSoft Inc., USA).

Linear partial least squares regression analysis (PLS) was used to analyze the relationships between sensory and physical matrices (15,16). PLS extracts a few linear combinations (PLS factors) from the physical data that predict as much of the systematic variations in the sensory data as possible. Because of the multivariate nature of the sensory data, PLS2 was performed (15), and all the sensory variables were correlated versus all the instrumental variables simultaneously. Osten's F-test (17) was used to determine the number of significant ($p \leq 0.05$) factors. The Genstat statistical language Release 4.1, Numerical Algorithms Group (18) was used for these analyses.

RESULTS AND DISCUSSION

ANOVA showed that between-repetition and between-assessor variations were not significant ($p > 0.05$), and neither were assessor-repetition, assessor-sample, and repetition-sample interactions. ANOVA showed all parameters (sensory and instrumental) to be significant in discriminating among samples ($p \leq 0.001$), results that support the validity of the chosen descriptors in developing the sensory profile.

INSTRUMENTAL DATA PRINCIPAL COMPONENT ANALYSIS (PCA)

In the PCA of the instrumental data for the eight samples, the first two principal components (PC) accounted for 62.6% and 34.4% of the variance, respectively. The first PC (PC1) contrasted surface tension (positively) with spreadability at one-half minute (S0.5) and one minute (S1) (negatively). The second PC (PC2) was defined negatively by viscosity. The emollient scores for the first two PCs are plotted in Figure 1.

Coordinate analysis enabled the discrimination of different groups of emollients according to the evaluated physicochemical properties. CM scored to the left side of the first PC, showing high values of spreadability at one-half minute and one minute and low values of surface tension. DM scored low in PC2, showing high values of viscosity.

A group, including SO, MO, and SQ, scored to the right side of PC1, showing high values of surface tension and spreadability at one-half minute and one minute. The group formed by IPM, DO, and OD showed intermediate properties.

The two silicones showed a different behavior. The high viscosity of DM did not result in difficulty of spreading under the action of gravity, as could be predicted, showing similar values of spreadability and surface tension to those of OD or MO. CM showed unique characteristics: its spreadability was approximately twice that of the rest of the emollients.

SENSORY DATA PRINCIPAL COMPONENT ANALYSIS

The first principal component accounted for 59.7% of the variance, with a positive

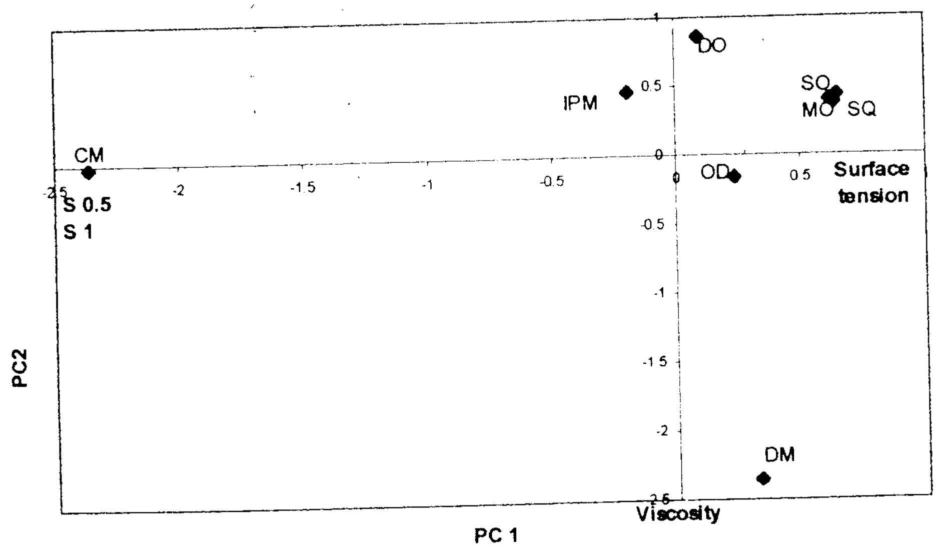

Figure 1. Instrumental data: Principal component analysis.

contribution of gloss, residue, and oiliness. The second principal component accounted for 36.3% of the variance, with a positive contribution of slipperiness and softness and a negative contribution of stickiness and difficulty of spreading. The emollient scores plotted for the first two PCs are shown in Figure 2.

The sensory evaluation data separated the studied emollients into four groups with different sensorial characteristics. CM scored to the left side of PC1, having a unique sensory profile among the selected emollients, showing low values of gloss, residue, and oiliness, due to the fact that it is a volatile product that leaves little residue on the skin. DM scored low in PC2, showing high values of difficulty of spreading and stickiness and low values of slipperiness and softness. Its sensory profile also differentiates it from the rest of the emollients. This could be attributed to its chemical composition (polydimethylsiloxane), as it showed the highest values of viscosity among the selected emollients.

The group formed by SO, MO, OD, SQ, and DO scored to the right side of PC1, showing high values of gloss, residue, and oiliness. Despite the different chemical structures of these emollients, their sensory profiles were very similar. IPM scored to the left side of PC1, in an intermediate position.

The correlation between sensory attributes showed that difficulty of spreading, stickiness, slipperiness, and softness are related. This is in agreement with the fact that these properties depend on the interaction between the forearm skin on which the product is applied, the skin of the hand that evaluates the product, and the product itself. On the other hand, gloss, residue, and oiliness are related, and these three attributes depend on the characteristics of the emollient film formed over the skin after application.

PLS

Leverage-corrected residual showed optimal prediction ability for two PLS regression

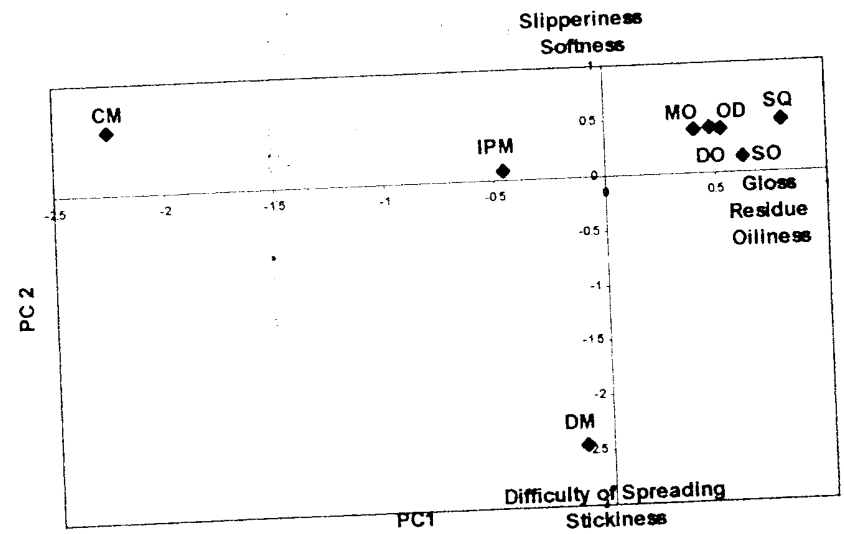

Figure 2. Sensory data: Principal component analysis.

factors, explaining about 87.9% of the total variance in y. For the individual descriptors, percent variance of experimental values accounted for by PLS Factor 1 and Factor 2 are shown in Table II. The correlation coefficients of sensory descriptors and instrumental measurements with the two PLS factors are presented graphically (Figure 3). Table II and Figure 3 show the PLS correlation to be bi-dimensional. This was confirmed by Osten's F-test, which determined that Factor 1 and Factor 2 were valid predictions ($p < 0.001$).

Table II shows that all attributes were well predicted by PLS regression on the instrumental parameters. Figure 3 shows that gloss, residue, and oiliness were positively correlated to instrumental surface tension and negatively correlated to spreadability at one-half minute and one minute. Instrumental viscosity was positively correlated with difficulty of spreading and stickiness and negatively correlated with softness and slipperiness.

These results suggest that sensory attributes related to mechanical instances of application or evaluation, such as difficulty of spreading, stickiness, and slipperiness, are related to physicochemical properties such as viscosity, that is, emollient resistance to flow and the forces needed to achieve it. Besides, sensory properties related to the film that the product leaves on the skin (gloss, oiliness, and residue) are related to physicochemical properties such as spreadability at one-half minute and one minute, and surface tension, properties that depend on the capacity of the emollient to develop films.

CONCLUSIONS

In considering their physicochemical characteristics, the studied emollients were sorted into three groups, in which the two silicones are distinctly separated from the rest. The sensory characteristics enabled the discrimination of four groups of emollients, where, besides the two silicones, IMP also differentiates.

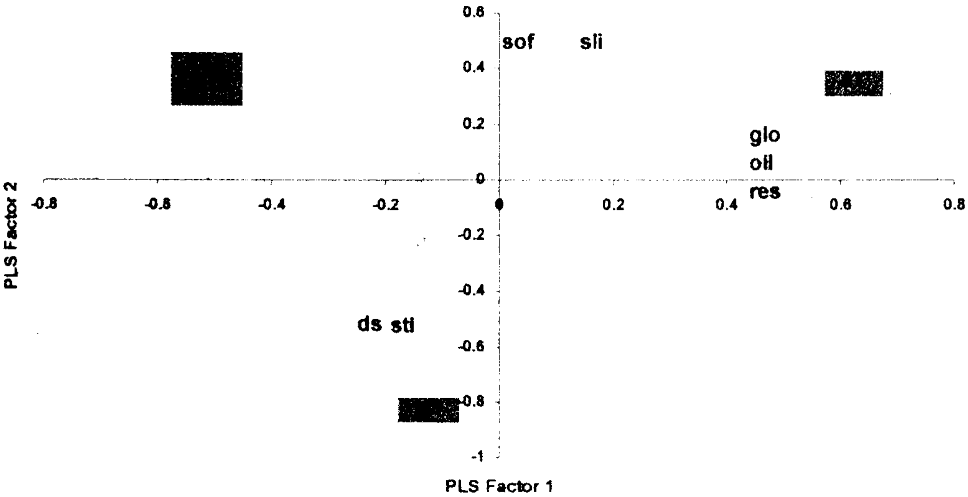

Figure 3. PLS2 loadings for the seven sensory variables (y-block) and four instrumental variables (x-block) analyzed on the eight samples. ds: difficulty of spreading. glo: gloss. res: residue. sti: stickiness. sli: slipperiness. sof: softness. oil: oiliness. S0.5: spreadability at one-half minute. S1: spreadability at one minute. ST: surface tension. VIS: viscosity.

Table II
Percent Variance of Experimental Values Accounted for by First Two Partial Least Squares Factors for Sensory Descriptors

	Factor 1	Factor 2
Difficulty of spreading	5.6	88.6
Gloss	81.8	5.9
Residue	90.8	1.4
Stickiness	0.1	95.6
Slipperiness	12.3	78.1
Softness	0.1	62.6
Oiliness	84.0	0.2

PLS revealed that emollient sensory attributes could be well predicted by instrumental measurements. Gloss, residue, and oiliness were correlated with surface tension and spreadability at one-half minute and one minute, whereas instrumental viscosity was correlated with difficulty of spreading, stickiness, softness, and slipperiness.

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