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Sensory analysis of cosmetic powders: personal care ingredients and emulsions

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Abstract

OBJECTIVE: The powders are ingredients increasingly used in the formulation of cosmetic products for the sensory qualities they give. The objective of this study was the development of a lexicon and a referential for sensory characterization of these pure raw materials as well as formulations which contain them.

METHODS: Eleven expert panellists from Ecole de biologie industrielle de Cergy (France) developed a lexicon and a referential based on 12 powders of different chemical natures. The selected attributes were then used for performing a quantitative descriptive profile of two powders and an emulsion containing or not one of these two powders.

RESULTS: A lexicon has been established through a consensus approach of the panel. It contains seven attributes that allow the evaluation of the powders in four phases: the appearance, the pickup, the application and the after-feel. This lexicon contains definitions and assessment protocols and provides references products. The quantitative descriptive profile of two powders of the same chemical nature, but different in physical quality showed significant differences in sensory level between products. These same attributes used to evaluate an emulsion containing the powder or not allowed to prove the contribution of these raw materials on the sensory specificities of the emulsion.

CONCLUSION: The lexicon developed in this study can be used for assessment of other powders but also to define the quantities necessary to put in the formulation to meet the sensory characteristics of these raw materials powder.

Résumé

OBJECTIFS: les poudres sont des ingrédients de plus en plus utilisés dans la formulation des produits cosmétiques pour les qualités sensorielles qu'elles apportent. L'objectif de cette étude, est le développement d'un lexique et d'un référentiel pour la caractérisation sensorielle de ces matières premières pures mais aussi des formulations qui les contiennent.

METHODES: 11 panélistes experts de l'Ecole de biologie industrielle de Cergy ont développé un lexique et un référentiel en se basant sur 12 poudres de natures chimiques différentes. Les attributs sélectionnés ont ensuite été utilisés pour la réalisation d'un profil descriptif quantitatif sur deux poudres et sur une émulsion base qui contenait ou pas l'une de ces deux poudres.

RESULTATS: Un lexique a pu être établit grâce à une approche par consensus du panel. Il contient 7 attributs qui permettent l'évaluation des poudres en 4 phases: l'apparence, la prise, l'application, après application. Ce lexique contient les définitions, les protocoles d'évaluation et propose des références. Le profil descriptif quantitatif réalisés sur deux poudres de même nature chimique mais de qualité physique différente a montré des différences significatives au niveau sensoriel entre les produits. Ces mêmes attributs utilisés pour évaluer une émulsion qui contenait ou pas les poudres a permis de monter l'apport des matières premières sur les qualités sensorielles de l'émulsion.

CONCLUSIONS: Le lexique développé dans cette étude peut être utilisé pour l'évaluation d'autres poudres mais aussi pour définir les quantités nécessaires à mettre dans la formulation afin de retrouver les spécificités sensorielles de ces matières premières pulvérulentes.

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Introduction

It has become essential for the cosmetic industry to respond to consumers' needs. Whereas the efficiency and safety of the products is indispensable, the sensory characteristics can improve consumer acceptability and sales of products. This is why ingredients are developed not only for technical functions but also for specific sensory targets. Among all these ingredients, powders represent a specific category widely used in make-up. Currently, these substances have become standard for the formulation of powder products especially in compacted specialty where they provide cohesion and a soft after-feel. In other segments (like skincare and sun care), powders can improve the performance and enhance the feel of the skin. For instance, the presence of talc causes an absorbent effect on the formulation and gives a non-sticky touch. Modified starches improve the texture and the viscosity of products and leave a soft after-feel. Nylon 12 (polyamide) combines a soft touch with good absorption properties and facilitates the application and spreadability. Unfortunately, all the above-mentioned claims are poorly documented, and, in general, there is a lack of scientific approach concerning the evaluation of ingredients on the skin-feel. In this field, the literature essentially concerns emollients. In 2005 and 2008, Parente *et al.* [1, 2] demonstrated that it is possible to quantify the skin-feel of emollients using a sensory quantitative descriptive profile method. Considering their sensory characteristics, the evaluated emollients were significantly sorted into groups related to the difficulty of spreadability, gloss, residue, stickiness and oiliness. Later, with the same approach, Lukic *et al.* [3] evaluated four emollients based on their sensory attributes: spreadability, texture, slipperiness, persistence of emolliency. In this study, they showed specifically sensory characteristics for each tested sample and a correspondence with the sensory data of a formulated cream. Recently, Savary and *et al.* [4] have performed a quantitative sensory evaluation on the spreadability of five oils and the spreadability of the five corresponding emulsions. Finally, they demonstrated that the emollients contributed a sensory significant effect to the emulsions.

In the field of cosmetic powders, Timm *et al.* [5] carried out research on the perceived skin-feel of powders in a suspension (Nylon 12 and PMMA). Several sensory attributes, such as powdery, silky and velvety, were generated and used by panellists to describe the sensory profile of each powder studied. The main objective of this work was to compare the results obtained with the panel and a measuring instrument dedicated to the evaluation of the powders' friction coefficient.

A real need has appeared to characterize powders with discriminant sensorial attributes because these ingredients are widely used for their sensorial properties in cosmetic products. The tactile perception of skincare products is normally evaluated by trained panellists. The quantitative descriptive profile method is the conventional approach for obtaining discriminating and repeatable results of a products' tactile performance [6]. The method is commonly used for the assessment of skin-feel during and after application of cosmetic products [7, 8]. The use of panels as instruments depends on the calibration and validation; well-defined and documented lexicons support this level of sensory research. Furthermore, sensory scientists need effective communication tools to relate their studies to non-technical business audiences. Lexicons establish the vocabulary that enables all of these entities to communicate.

The main purpose of this study was to develop a sensory protocol of evaluation for pure powders and for emulsions containing these ingredients. The first objective is to obtain a lexicon and a referential adapted to the calibration and validation of a panel. The second objective is to evaluate the influence of the presence of the powders on the sensorial properties of the formulated products.

Materials and methods

Materials

Products

The sample set should be large enough to provide a fair representation of the entire product category. In this study, thirteen powders were used to generate sensory attributes (Table I). Two other products (PSA and PSB) were assessed by a quantitative sensory descriptive profile. The PSA and the PSB differed in their particle size distribution, between 2.5 and 5.5 μm (D50 = 4.00 μm) and between 2 and 8 μm (D50 = 3.98 μm), respectively, as shown in Figs 1 and 2.

The selected powders differ in their chemical nature (Table I). There are mineral powders (talc), chemical compounds (derivatives of siloxanes) and mixtures of powders (mineral, vegetable, chemical compounds).

An emulsion was also developed to evaluate the sensory impact of two powders (PSA and PSB) on formulations (Table II).

Table I Raw materials used for protocol development and sensory evaluation

Code	INCI name
GLS	Magnesium silicate and triethoxycaprylylsilane
GLD	CI 77891 & CI 77491 & Mica & triethoxycaprylylsilane;
MAT	Mica and talc and titanium dioxide and lauroyl lysine
BRN	Boron nitride
SLC	Silica dimethyl silylate
DTS	Tapioca Starch (and) Polymethylsilsesquioxane
RSP	Calcium Aluminium borosilicates, CI 77881 (Titanium dioxide), silica, tin oxide
TPE	Talc
OPA	Titanium dioxide (and) alumina
ARG	CI 77891 & CI 77491 & Mica & triethoxycaprylylsilane
PSA	Polymethylsilsesquioxane
PSB	Polymethylsilsesquioxane
PMS	Polymethylsilsesquioxane
PMA	Polyméthylmétacrylate

Prior to the sensory evaluation, the safety of the powders (Table I) and emulsions (Table II) was assessed for use on skin.

Panel

The panellist selection is important in all descriptive analysis, but particularly in the lexicon development. At EBI, a very large panel (almost 100 students) is trained in sensory analysis (taste, touch and vision) on different products over a period of 6 months. Eleven engineering students at the Ecole de Biologie Industrielle were selected to be on this panel based on their motivation.

In accordance with ISO 8589 [9], the panel worked in an standardized environment. A dedicated test room isolated from external disturbances, with temperature and humidity control, was used. The sensory evaluations took place in individual booths with homogenous artificial lighting.

Methods

The sensory quantitative descriptive profile was the method selected to perform the evaluation of cosmetic products and ingredients. The profile is obtained by the statistical processing of data from multiple subjects using a single list of attributes [10].

Lexicon and references

Different steps were necessary to develop the lexicon of powders: first generating terms, secondly, reducing the list and defining the attributes and thirdly identifying the references.

- Generation of terms: The sample set should be large enough to provide a fair presentation of an entire product category. In this study, thirteen powders (raw materials, presented in Table I) were given to the panellists. During four 1-h sessions, they had to generate all of the words that describe sensations provided by the products. After these four sessions, four lists were obtained. In accordance with ISO 11035 [11] (identification and selection of attributes for establishing a sensory profile by a multidimensional approach), hedonic terms were eliminated from these lists.
- Reduction and definition: Relevant sensory attributes, quoted at least twice, were selected in the resulting lists. After this

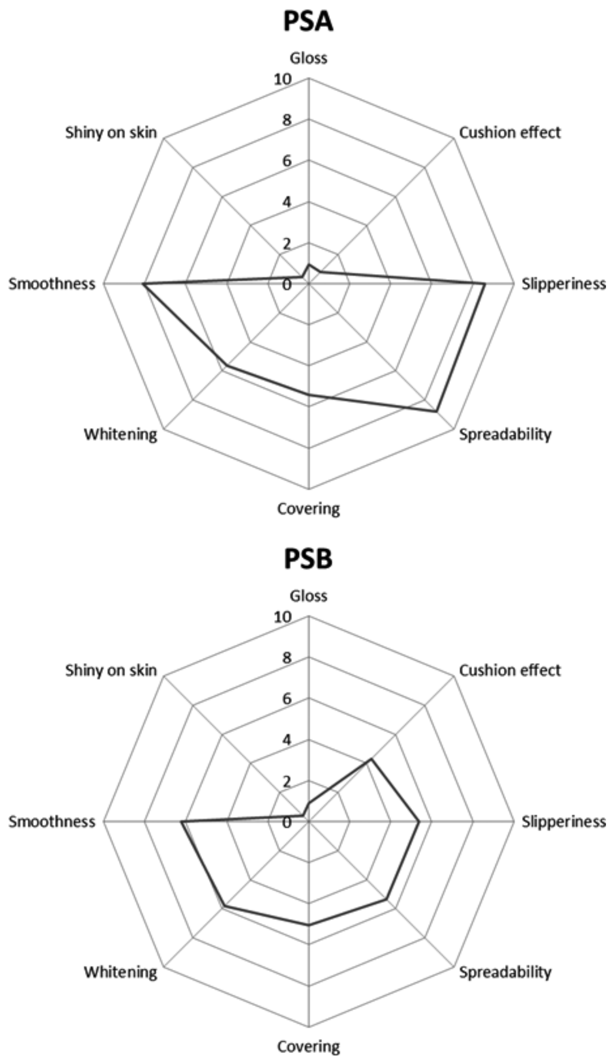

Figure 1 Radar of PSB and PSA obtained by the method of quantitative sensory profile.

selection, two sessions were necessary to develop the terminology and the protocol of evaluation thanks to a consensual approach. Finally, a 4-phase protocol of evaluation was chosen, as detailed in Table V. These phases were based on the appearance in the jar, pickup between thumb and forefinger, application on skin and evaluation of after-feel.

- Identification of Reference: As scientific literature could not reveal helpful references for attributes, it was necessary to identify adapted powders. A procedure was carried out to determine the reference's appropriateness. To this end, each panellist evaluated the potential references individually for each attribute on a structured scale from 0 to 10. When a powder was quoted with a high score (close to 10), it was proposed as the reference for the attribute. In contrast, when it was quoted with a very low score, it was proposed as the minimum reference for the attribute.

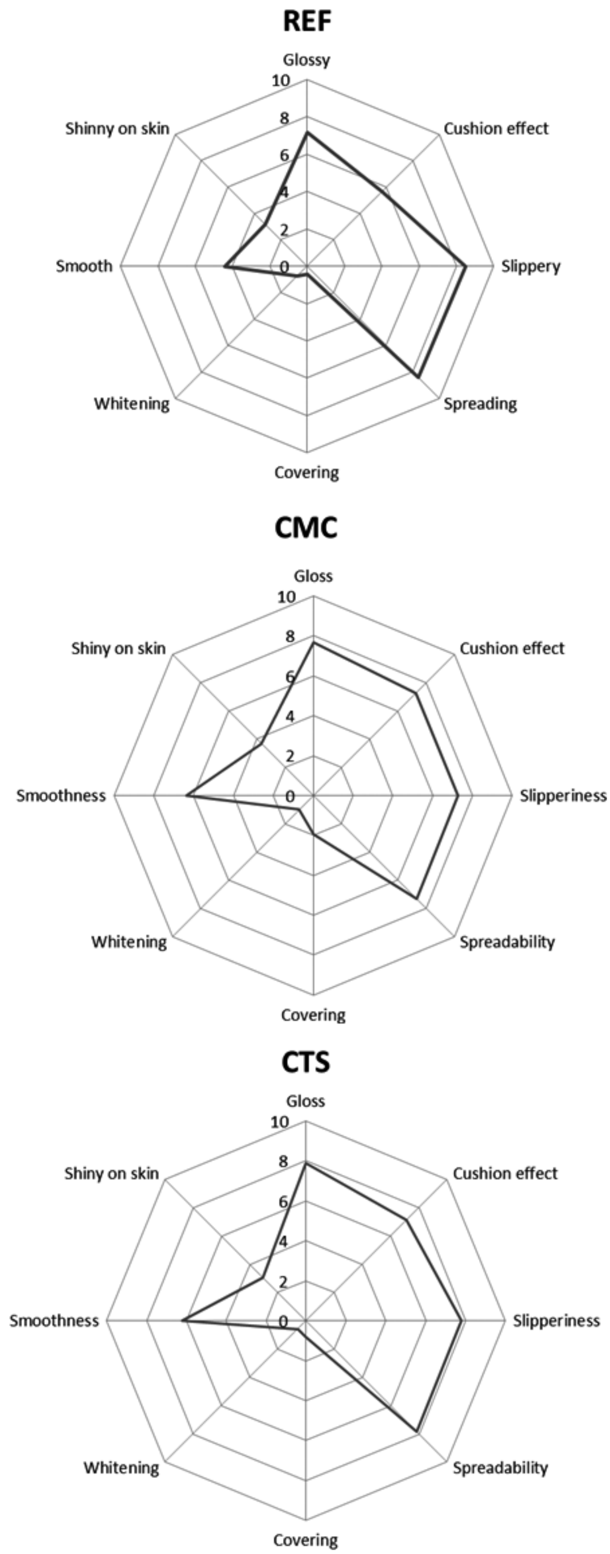

Figure 2 Radar of emulsions CMC, CTS and REF obtained by the method of quantitative sensory profile.

Table II Formulations used for sensory evaluation

INCI name	REF	CTS	CMC
Aqua	76.65%	70.65%	70.65%
Isopropyl myristate	10%	10%	10%
Polymethylsilsesquioxane (PSA)	–	5.5%	–
Polymethylsilsesquioxane (PSB)	–	–	5.5%
Decamethylcyclopentasiloxane	5%	5%	5%
Glycerin	3%	3%	3%
Potassium cetyl phosphate	1.5%	1.5%	1.5%
Magnesium aluminium silicates	1.5%	1.5%	1.5%
Cetyl alcohol	1%	1%	1%
Phénoxyéthanol	0.9%	0.9%	0.9%
Glycerol monostearate	0.5%	0.5%	0.5%
Xanthan gum	0.15%	0.15%	0.15%
Potassium sorbate	0.1%	0.1%	0.1%

Sensory profile

A balanced and randomized experimental design was used for evaluation. Each product (Tables I and II) was assessed only once per session. Three sessions were necessary for powders and two sessions for emulsions. The samples were presented to panellists in a sequential monadic design, to avoid bias of measurement. The evaluation protocol consisted of four steps:

1 The appearance of each product (powder or emulsion) was evaluated in the jar, under the light of the booth.
2 Each product (powder or emulsion) was assessed between thumb and forefinger, for the pickup phase.
3 Each product was spread in ten circular movements of a 2 cm diameter, on top of the hand to evaluate application.
4 The tip of the finger was rubbed on top of the hand, where each product had been previously applied, to evaluate the after-feel.

Statistical analysis

Using the analysis of variance (ANOVA), the discriminating power of the panel was assessed [11]. Samples were compared using the Kruskal–Wallis test and the Tukey's HSD test. Microsoft Excel (Excel 2013 version 15.0.4727.1003 and Excel 2007) and XLstat® (version 2014.6.5) software was used to perform the analysis.

Results and discussion

Lexicon and references

Descriptive sensory methods are among the most sophisticated tools in a sensory scientist's domain to describe the qualitative and quantitative sensory attribute of a consumer product [11, 12]. With this method, subjects should assess products following a common list of sensory attributes. This list can be either pre-established or constituted by a group of panellists under the direction of a manager. In this study, the method followed the steps below [13, 14]:

1 Collected 13 powders (Table II)
2 Generated terms for the panel in contact with the samples (four 1-h sessions over four consecutive weeks)
3 Shortened the list

Table III Lists of terms given by Panellists during the lexicon generation

Session 1	Session 2	Session 3	Session 4
White	White	White	White
–	Gold	–	–
–	Pink	–	–
Fine powder	Fine powder	Fine powder	Fine-PMSined
–	Particles' size	–	PMSins' size
Visible PMSin	–	–	PMSins
–	–	–	Powders' aggregates
–	–	–	Aggregates size
Aggregates	–	–	Aggregates
Residue	Residue	Residues on skin	
Absorption	–	–	Penetrating
–	Absorption in pores	Penetrating in pores of skin	Impregnation in pores of skin
–	–	–	No residue
–	Homogeneous	–	–
Easy to spread	Easy to spread	Easy to spread	Easy to spread
Resistance spreadability	–	–	Brake spreadability
Shiny on skin	Shiny on skin	Shiny on skin	Shiny on skin
Pearly on skin	–	–	Pearly on skin
Silk finish (visual) (visual)	–	–	Silk finish
–	–	–	Mattifying
Smoothness	Smoothness	Smoothness	Smoothness
–	–	–	Rough
Gloss	Gloss	Gloss	–
Mate	–	–	–
Pearl	–	–	–
Blur (near blusher effect, soft focus effect)	–	–	–
Opaque	–	–	–
Flow	–	Flow	Flow
–	–	–	Compact
Volatile	–	–	Volatile
Dry	–	–	Dry
–	–	Perceived/not perceived PMSins	Perceived PMSin
–	–	Cushion effect	Cushion effect
–	–	Whitening	Whitening
–	–	Skin coloured by product	–
–	–	–	Covering
–	–	Slipperiness	Slipperiness
–	–	–	Homogeneous, PMSins fall during spreadability

4 Developed a definition for each remaining sensory attribute
5 Proposed references for each attribute

During step 2, four lists were obtained. Looking at the results (Table III), an evolution was observed where by the collected number of terms and their precision improved from session to session. The first list gathers twenty terms, but some of these terms were not directly in connection with the product space (e.g. pearly and blur). Twelve descriptive attributes were obtained during the next session. Fifteen sensory attributes were then generated during the third session. In the final session, thirty sensory attributes were obtained.

Table IV Sensory attributes, definitions and references in products' space (powders and emulsions with powders

Sensory attributes	Definition	Maximum limit (10)	Minimum limit (0)
Appearance			
Gloss	Amount of light reflected of product in the jar	RSP	MAT
Pickup			
Cushion effect	When the product is between thumb and forefinger, the movement is dampened.	RSP	BRN
Slipperiness	Ease of moving between thumb and forefinger	TPE	SLC
Application			
Spreadability	Ease of moving the product over the skin	PMS	MAT
Covering	Degree product covers the imperfections of the skin	GLD	SLC
Whitening	Degree product turns white when rubbed	BRN	SLC
After-feel			
Smoothness	Degree skin, not marked by roughness	TPE	SLC
Shiny on skin	Amount of light reflected of skin	RSP	DTS

In accordance with ISO 11035 [12], sensory attributes must be relevant, discriminant, specific, independent and exhaustive. Terms that did not meet these specifications were eliminated. The final shortlist was established based a consensual decision by groups. For instance, panellists from both groups arranged together synonyms and opposite terms (e.g. spreadability and breaking effect). In addition, the most cited terms did not all make it to the final list (e.g. white).

This step results in a consensual list of eight sensory attributes. After this step, two sessions (1 h per session) were necessary to define references and definitions of each sensory attribute (Table IV).

Method of sensory profile

The aim of our work was to obtain a sensory profile of two powders and to characterize their influence on emulsions. Powders and emulsions were assessed, and the panel's performance was controlled using the ANOVA (with product effects and session effects). The ranking test (Kruskal–Wallis) was used to validate the results of ANOVA. At the beginning of each session, feedback on the previous session was given to improve performance.

At the end of the sessions, a final ANOVA and the Kruskal–Wallis test were performed for the data set concerning the powder samples (in Table V) as well as for the data set concerning the emulsion samples (in Table VI). This analysis evaluated the panel's performance thanks to the *F*-test of product effect. When the *P*-value is lower than 5%, the discrimination is validated.

For powders (Fig. 1, Table V), four discriminant attributes were identified (at 5% level of significance), which were cushion effect, slipperiness, spreadability and smoothness. In addition, some non-significant differences might have been masked by a lack of consensus between panellists. So, even if there was some bias between the panellists, the ranking test should be able to detect any differences between products. In this study, we used a standard ranking test: the Kruskal–Wallis test, where all *P*-values are presented in Tables V and VI. For the powder samples, the ranking test identified the same discriminating attributes as the *F*-test.

Table V For each sensory attribute, *P*-values of the product effect (Pr > F) from the ANOVA of powders and *P*-value from Kruskal–Wallis test (Pr > K) (* when Pr < 0.05).

Attributes	Pr > F	Pr > K-
Gloss	0.856	0.518
Cushion effect	<0.001*	<0.001*
Slipperiness	<0.001*	<0.001*
Spreadability	<0.001*	<0.001*
Covering	0.585	0.651
Whitening	0.793	0.726
Smoothness	<0.001*	<0.001*
Shiny on skin	0.721	0.920

Table VI For each sensory attribute, *P*-values of the product effect (Pr > F) from the ANOVA of emulsions and *P*-value from Kruskal–Wallis test (Pr > K) (* when Pr < 0.05)

Attributes	Pr > F	Pr > K-
Gloss	0.284	0.192
Cushion effect	0.022*	0.041*
Slipperiness	0.326	0.251
Spreadability	0.075	0.128
Covering	0.124	0.030*
Whitening	0.566	0.272
Smoothness	<0.001*	0.004*
Shiny on skin	0.640	0.638

For emulsions (Fig. 2, Table VI), the differences between the emulsions are significant at 5% for two of the attributes which were covering and smoothness. The spreadability attribute accounted for 10%. Referring to the ranking tests, we can see that the spreadability attribute is not validated as significant. In addition, the covering attribute is significant for the ranking test but not for the *F*-test. We can see in the radar plots, in Fig. 2, that the covering scores are very low and are therefore insignificant.

To analyse the differences, we used a standard pairwise test: the Tukey's honest significant difference (HSD) test. The results are presented in Tables VII and VIII. Some products are associated with the same letter which means that the products are not significantly different (at 5% level of significance). However, when the associated letters are different, it means the samples are significantly discriminating (at 5% level of significance).

When looking at the results for powders, we can see in Table VII that the powders are similar except for the cushion effect, slipperiness, spreadability and smoothness. The PSA has less of a cushion effect than the PSB, and on the other side, the PSA has more slipperiness and more spreadability and is smoother than PSB.

When looking at the results for emulsions in Table VIII, the sensory differences are restricted to the cushion effect and the

Table VII Means of the powders samples with Tukey's HSD pairwise test at the 5% significance level.

Attributes	PSA	PSB
Gloss	0.939 a	0.879 a
Cushion effect	0.818 b	4.333 a
Slipperiness	8.576 a	5.364 b
Spreadability	8.818 a	5.364 b
Covering	5.394 a	5.061 a
Whitening	5.636 a	5.788 a
Smooth	8.091 a	6.212 b
Shiny on skin	0.455 a	0.394 a

Table VIII Means of the emulsions samples with Tukey's HSD pairwise test at the 5% significance level.

Attributes	CMC	CTS	REF
Gloss	7.682 a	7.909 a	7.167 a
Cushion effect	7.273 a	7.136 a	5.667 b
Slipperiness	7.27 a	7.773 a	8.500 a
Spreadability	7.318 a	7.864 a	8.417 a
Covering	1.955 a	0.818 a	0.417 a
Whitening	1.000 a	0.591 a	0.750 a
Smooth	6.367 a	6.227 a	4.417 b
Shiny on skin	3.682 a	3.045 a	3.167 a

smoothness. There are no differences between the two mixtures with the powders, but there are differences between the basic formula and the two emulsions with 5.5% of powder. The cushion effect and the smoothness increase significantly (at 5% level of significance) when we add the powders to the emulsions.

Discussion

A lexicon of 8 attributes (Table IV) was developed to describe the visio-tactile characteristics of cosmetic powders. From this list of attributes, half of them describe specific sensory characteristics related to their use in make-up (e.g. gloss, covering, whitening, shininess on the skin). The other half characterizes their texture on contact with the skin (e.g. slipperiness, spreadability, cushion effect, smoothness), which are desirable for cosmetic application. Of these attributes, only the 'texture' attributes were discriminant for the evaluation of two marketed powders of polymethylsilsesquioxane. The ANOVA of these powders (Table V) and the pairwise comparisons (Table VII) showed significant differences for four sensory attributes in the pickup, application and after-feel phases. The scores of products for each sensory attribute are presented as a radar in Fig. 1. The PSB (4.33) had a higher score for cushion effect than the PSA (0.818). The slipperiness, spreadability and smoothness scores of the PSA (8.576; 8.818 and 8.09, respectively) were higher than the PSB (6.758; 5.364 and 6.212, respectively). There was no significant difference in appearance between the PSA and the PSB. The powders have the same INCI denomination, so there should have been no significant differences. This was, however, not the case. Dissimilarities between the powders could be explained by the repartition of the particle size of each powder (PSA and PSB have the same medians, the standard deviation of the particle size of PSA is 0.53 and the standard deviation of PSB is 1.07). The larger the range of the particles, the more easily they even out the skin's asperities. In addition, when the range of particles is larger, there is a roll on effect when applied. In accordance with these results, the score of slipperiness, spreadability and smoothness attributes is higher when the range of particles' size is large. The score of the cushion effect attribute is positively correlated with the particle's size repartition.

Looking at the pairwise comparison for emulsions in Table VIII, we can see that the two emulsions with powders (5.5% w/w) were not significantly different. Referring to the work of Timm *et al.* [5], the authors found that the quantity of powder has to be higher than 10% of powders in the suspension to detect a difference.

Unlike Timm *et al.* who worked on powders into suspension, we worked on emulsions with 5.5% of powders (see Table II, for the all formulations).

Looking at the emulsions without any powder (REF – Table VIII), the statistics show that there were significant differences between samples with powders and samples without any powders for the following attributes: cushion effect and smoothness. These dissimilarities prove that the addition of powders lead to sensory modifications of the emulsion (5.5%). In accordance with the results, the scores of cushion effect and smoothness will increase when we add some powders. However, the impact of differences between both powders was not relevant for gloss, slipperiness, spreadability, covering, and whitening and shiny on skin with a concentration of 5.5 % of powder.

Conclusion

A protocol of sensory evaluation was developed to characterize pure cosmetic powders and their impact on emulsions. A collective effort using a qualified group of people was necessary to develop the lexicon and definitions [8]. Our panellists were able to characterize products and provide sensory profiles that are relevant to assess sensory profiles for a collection of attributes about appearance, pickup, application and after-feel phases.

In this study, we worked on two powders with the same INCI denomination, but with different distributions of particle sizes. We found that there were significant differences between these products concerning the cushion effect, slipperiness, spreadability and smoothness attributes. These differences could be explained by a better repartition of the particles on the skin's asperities when the range of particles size is larger.

We also have shown that the addition of powders (at 5.5% of concentration) to the formulation increases the cushion effect and the smoothness of the emulsion. In addition, despite the differences between the two powders (with the same INCI denomination), we found that the two powders have similar effects in an emulsion when they are added at 5.5% of concentration.

In the future, improving the sensory method and working on the effect of powders in emulsions could be examined using two different approaches, for example enlarging the product space with a wide range of cosmetic powders to validate the use of the lexicon and carrying out an experimental design of formulations varying the quality and quantity of both powders and emulsions.

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