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# Spreading behavior of cosmetic emulsions: Impact of the oil phase

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## ABSTRACT

*Background*: Emollients are an important ingredient in personal care products, they are currently prescribed in skin disorders like eczema, which affects up to 30% of children in developed countries. *Methods*: The aim of the study was to investigate the impact of two emollients (stearic acid and isohexadecane) and their mixtures in the spreadability and frictional effect on films obtained from oil-in-water emulsions. Rheological, textural, sensory and tribological analysis were performed on human skin and artificial substrates. *Results*: The emollients ratio influences the spreading behavior of emulsions: more isohexadecane in the oil phase easier to spread the product on the skin. Moreover, significant correlations were obtained for the spreading behavior obtained by textural measurements on artificial substrates and sensory analysis (Pearson coefficient = -0.871). The results obtained by frictiometer showed different developments over time after product application: the friction values increase with the stearic acid concentration in emulsion. *Discussion*: This study showed the importance to consider the emollient properties when one emollient is used in the emulsion, but especially their interactions, when several emollients are used, to better understand and anticipate their behavior. First, spreading was governed by the consistency of the emulsion, particularly impacted by the emollients ratio. But then, in a long-time spreading, when the emulsion broke down and residual film was

formed, a particular interaction with skin influenced the spreadability. It appears that not only the physical state of the emollient but also its chemical nature, physical state, polarity, temperature might explain these phenomena.

#### 1. Introduction

Emollients are multifunctional ingredients supporting multiple formulation claims in cosmetology, dermocosmetology and dermatology [1–3]. For instance, emollient therapy is currently prescribed in managing eczema (or atopic dermatitis), since this skin disease can affect up to 30% of children in developed countries [4–6]. In skin care emulsions, emollients represent the major ingredient after water, being used at level between 3 and 20% (*w*/w) [7]. From a sensory perspective, emollients have a major impact on physicochemical properties of cosmetic emulsions such as consistency and spreadability, properties that are important to achieve adequate efficacy and user acceptance of the products [8]. Generally, during application on the skin, emollients decrease the friction coefficient of the emulsion due to their lubricant properties and modify its spreading performances [9]. Therefore, spreadability is one of the sensory characteristics commonly evaluated during emulsion application [10].

Spreading can be defined as the ability of a substance to cover a surface and depends on molecular weight, viscosity and chemical structure [7,11]. In general, constituents with low molecular weights

and/or low viscosities have higher spreading properties. Thus, the choice of emollients is essential to control the efficacy of the product in terms of skin moisturizing, but also to achieve the satisfactory physical and chemical stability of the emulsion. In particular, the polarity of the emollients and the association of different ingredients affect the mechanism of interactions with the skin as well as the structural organization and organoleptic characteristics of the emulsion [12]. In this respect, some studies attempted to characterize different pure emollients for their spreadability properties using rheology, spreading value and contact angle measurements on synthetic substrates and sensory analysis [8,11,13-16], but very few studies focused on the impact of emollients on the spreading properties of complex emulsions and creams [17,18]. Moreover, to the best of our knowledge, no study has been devoted to the mixture of different emollients incorporated in complex emulsions. In this way, the first aim of our study was to observe the effect of two types of emollients (pure and their mixture), commonly used in the cosmetic field: a close to solid saturated fatty acid - the stearic acid (SA) and a mineral oil - the isohexadecane (IHD), on the spreading properties of cosmetic emulsions. These emollients were selected for their chemical structure and for their physical state,

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which may influence the consistency of the final product. Another target of this study was to establish how the consistency of the emulsions impacts the friction behavior on the skin.

The skin is a critical interface between the human body and its surrounding environment which accomplishes multiple defensive and regulatory functions. In particular, the 10-40 µm outermost thick layer of skin, namely the stratum corneum, by its complex multicomponent organization and its compact architecture, acts as the primary interface during application of skin care products [19]. At this stage, it seems important to understand the interaction between the skin and the emulsion. Recent reports focused on skin tribology, since it plays an important role while using skin care products in our daily life, being closely related to the contact and friction behavior between human skin and product surfaces [20-23]. These studies tried to understand and characterize the tribological properties of the bared skin, but very few concerned the friction evolution during the application of skin care products on skin [24,25]. Since sensory and tribological measurements on human skin are expensive, time-consuming and restrictive, it becomes important to have alternative strategies to understand this feature. Thus, our third aim was to explore the skin-emulsion interaction on the spreadability of cosmetic emulsions, by exploring different methodologies.

For this purpose, different methods were implemented in order to obtain a broader characterization of the spreading properties. Very few studies concerned the spreading of cosmetic emulsions [17,26] and, to our knowledge, no one reported a similar exhaustive work, including the use of a frictiometer and especially carried out on mixtures of emollients. Most of them only focused on correlations between sensory and rheological data [27,28] or tribological properties of naked skin [20,22]. The originality of this work was to fix the composition of the emulsion and to act on consistency only by varying the ratio of the two constituents of the oily phase.

In the present investigation, we have assessed how the nature of the oil phase of five O/W emulsions may impact their spreadability on the skin. By nature is meant the physical state of emollients and their ratio in the oil phase. For this purpose, two different emollients (IHD and SA) were incorporated at different rates (from 0 to 10%) in cosmetic emulsions. The impact of emollients and their ratio on the friction behavior of the O/W emulsions formulated with these oil mixtures was studied by a combined *in vitro* instrumental/*in vivo* study approach (texture analysis and rheology/sensory analysis and friction tests). These complementary tools made possible to better understand how emollients influence the spreadability of emulsions onto the skin.

#### 2. Materials and Methods

#### 2.1. Emulsions Preparation

Five O/W emulsions (200 g) were prepared by varying the composition of the oil phase in order to analyze the effect of two emollients: IHD and SA incorporated at different ratios (Table 1). Emulsions consisted in 10% oil phase, 5% emulsifiers and 85% aqueous phase. The oil phase and emulsifiers were first prepared by mixing and heating (75  $^{\circ}$ C) IHD (IMCD, France) and/or SA (Croda, France) with emulsifiers Steareth 2 and Steareth 21 (Croda, France) at 3 and 2%, respectively. The purified water (Qs) was heated at 75 °C. When all ingredients were at 75 °C, the oil phase and emulsifiers were added to the water under mechanical stirring (Turbo test, VMI, France) at 400 RPM for one minute. The emulsion was then homogenized at 11000 RPM during one minute using a T25 digital ultra-thorax (IKA, Germany) equipped with the rotor-stator turbine S25 N-25F. Each emulsion was continuously stirred using the Turbotest apparatus (VMI, France) until it cooled down to 40 °C. Then, 0.5% of cosmetic grade xanthan gum (Rhodicare® T, Rhodia, France) previously predispersed in 4% of butylene glycol (Across Organics, France) at room temperature under manual stirring. was added to the emulsion. At room temperature, water loss was compensated and 0.5% of preservatives Dekaben MEP® (Jan Dekker International, France) were incorporated and the emulsions were stirred for 15 additional minutes. The pH of the final emulsions was corrected by adding a sufficient amount of 1 M NaOH (CarloErba, Italy). The adjusted pH is indicated in Table 1.

The emulsions were stored at 4 °C to ensure preservation before further analysis. Their physical stability (at ambient temperature, 4 °C and 40 °C) in time was checked by particle size and rheological measurements (results not shown). The microbiological safety was assessed prior to sensory and *in vivo* analysis of the skin.

## 2.2. Microstructural Characterization

#### 2.2.1. Optical Microscopy

The microstructure of the Amazons was analyzed using a photomicroscope equipped with a camera (DMLP/DC 300, Leica Microsystems, Germany) at 200 x magnification for qualitative evaluation. The images were captured by the LAS V4.9 software (Leica Microsystems, Germany) on thin layers of emulsions on a microscope slide with a cover plate.

#### 2.2.2. Droplet Size Distribution

Droplets size measurements were performed by means of a laser diffraction particle size analyzer SALD 7500 Nano (Shimadzu Co., Ltd., Japan), equipped with a violet semiconductor laser (405 nm) and a reverse Fourier optical system. The emulsions were dispersed in purified water in order to reach an absorption value of  $0.200 \pm 0.10$ . To ensure homogenous dispersion of emulsions, continuous stirring was applied during analysis in the batch cell (7 cm<sup>3</sup>). All measurements were made at ambient temperature on at least three separately prepared samples. Data was collected using WingSALD II-7500 software.

#### 2.3. Instrumental Spreading Characterization

#### 2.3.1. Rheology

Continuous flow measurements were performed with a stress controlled rheometer (HR-1, TA Instruments, USA), using a cone-plate aluminum device (40 mm diameter, 1°59′38″ cone angle, 47  $\mu$ m gap) as described in previous work [18]. Here we only focused on shear-rate viscosities at 0.1 s<sup>-1</sup> which can be representative of the consistency of

#### Table 1

Ratio of isohexadecane (IHD): stearic acid (SA) in the oil phase (in %), pH, median particle size (D50) from granulometric measurements (in  $\mu$ m) and values of viscosity (in Pa.s) obtained at different shear rates (in s<sup>-1</sup>) obtained from shear flow test for the five O/W emulsions.

Emulsion	IHD:SA ratio	pH	D50	η(0.1)	η(1000)
I0SA10 12.5SA7.5 15SA5 17.5SA2.5 110SA0	0:10 2.5:7.5 5:5 7.5:2.5 10:0	$5.73 \pm 0.00 5.72 \pm 0.01 5.73 \pm 0.01 5.74 \pm 0.01 5.71 \pm 0.01 $	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$139.5^{b} \pm 2.2$ $167.5^{a} \pm 12.9$ $122.5^{c} \pm 2.6$ $102.2^{d} \pm 1.3$ $53.8^{e} \pm 0.9$	$\begin{array}{rrrr} 0.06^{\rm b} \pm 0.00 \\ 0.08^{\rm a} \pm 0.00 \\ 0.06^{\rm b} \pm 0.00 \end{array}$

a-e Means in a column sharing common superscripts are similar as tested by Tukey's test (P > .05). Means of replicates  $\pm$  SD.

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