



# Relationship between sensorial and physical characteristics of topical creams: A comparative study on effects of excipients

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## ARTICLE INFO

### Keywords:

Pickering emulsions  
Topical creams  
Tactile friction  
Sensory study  
Surfactant-free formulations  
Modified starch  
Rheology

## ABSTRACT

Rising consumer demands for safer, more natural, and sustainable topical products have led to increased interest in finding alternative excipients, while retaining functionality and cosmetic appeal. Particle-stabilized Pickering creams have emerged as possible alternatives to replace traditional surfactant-stabilized creams and are thus one of the focuses in this study. The aim of this paper was to study relationships between sensorial characteristics and physical properties to understand how different excipients affect these aspects, comparing one starch particle-stabilized and three surfactant-stabilized formulations. A human panel was used to evaluate sensorial perception, while physical properties were deduced by rheology and tactile friction, together with *in vivo* and *ex vivo* skin hydration measurements.

The results show that sensorial attributes related to the application phase can be predicted with rheology, while afterfeel attributes can be predicted with tactile friction studies. Differences in rheological and sensory properties among surfactant-based creams could mainly be attributed to the type of emollients used, presence of thickeners and surfactant composition. Differences between surfactant-based creams and a Pickering cream were more evident in relation to the afterfeel perception. Presence of starch particles in the residual film on skin results in high tactile friction and low perception of residual coating, stickiness, greasiness, and slipperiness in sensorial afterfeel.

## 1. Introduction

Development of cosmetically appealing topical formulations is important for both the cosmetic and pharmaceutical industry. Topical drug formulations are used for local treatment of various types of skin disorders but are also interesting for systemic delivery. The main advantage of topical delivery is avoidance of the hepatic first-pass metabolism, allowing local skin treatment, and decreasing the risks of side effects from some drug substances. Emulsions in the form of creams, lotions, gels or foams are the most common formulations for topical

delivery as they have good cosmetic properties and are suitable for delivery of hydrophobic substances (Marto et al., 2016; Wahlgren et al., 2013).

There is a constant demand for formulators to develop novel safe formulations of already existing patented drug substances (Wahlgren et al., 2013). Formulations need to meet constantly shifting consumer demands in terms of patient-compliance, cosmetic appeal (Lee and Maibach, 2006) and rising ethical demands by consumers (e.g. natural and sustainable), while still retaining the function intended. New formulation prototypes need to be analyzed for functional as well as

**Abbreviations:** Pickering emulsions, particle-stabilized emulsions; TEWL, *trans*-epidermal-water-loss; O/W, oil-in-water (emulsions); PC.c, Pickering cream with carbomer; SC.c, Surfactant-based cream with carbomer; SC, Surfactant-based cream;  $\mu$ S, micro Siemens; a.u, arbitrary units; LVR, linear viscoelastic region;  $G'$ , storage modulus;  $G''$ , loss/viscous modulus;  $\delta$ , phase angle; F, friction force; L, applied load;  $\mu$ , friction coefficient; LSD, least-significant-difference; PLS, partial least squares regression analysis;  $\chi^2$ , Chi-square.

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<https://doi.org/10.1016/j.ijpharm.2021.121370>

Received 27 August 2021; Received in revised form 27 November 2021; Accepted 7 December 2021

Available online 21 December 2021

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cosmetic properties.

Functional properties can be studied by physical measurements, while the cosmetic and sensory properties of topical products are normally evaluated by human panels. Panel-based sensory studies normally require a trained group of panelists or a large consumer panel and can be time-consuming and costly. Furthermore, human panels provide subjective evaluation of the products that is not always beneficial. The use of physical methods to screen new formulation prototypes for certain sensory attributes would be more suitable, allowing for more objective evaluations and major savings on time and costs. In addition, understanding how specific ingredients affect the physical and cosmetic properties relating to certain sensory attributes would be useful to help formulators develop products that meet consumer demands. Thus, there is a need to find physical methods which correlate with specific sensory attributes, and that can distinguish ingredients that cause a certain perceptible sensation. Several authors have in fact made successful attempts to connect physical characteristics of topical formulations to certain sensory attributes. Some of the methods that have been linked to sensory attributes are rheology (Greenaway, 2010; Huynh et al., 2021; Lee et al., 2021; Lukic et al., 2012; Savary et al., 2019; Vergilio et al., 2021), frictional analysis (Lee et al., 2021; Savary et al., 2019; Skedung et al., 2016; Timm et al., 2012) and texture analysis (Huynh et al., 2021; Lee et al., 2021; Savary et al., 2019; Vergilio et al., 2021). Rheology was found to be a useful tool to predict sensory attributes related to the application phase of cosmetic emulsions (Lee et al., 2021; Savary et al., 2019), while texture analysis was found to be a good predictor of textural attributes comprising stickiness and firmness (Lee et al., 2021; Savary et al., 2019). Some authors also found correlation between rheological and textural analysis (Gilbert et al., 2013; Lukic et al., 2012; Vergilio et al., 2021). Frictional analysis has been studied to a lesser degree but has been found to be a good predictor for afterfeel attributes (Lee et al., 2021; Savary et al., 2019). Recently, Skedung et al. (Skedung et al., 2016) developed a method to more accurately measure tactile friction of topical formulations by sliding the fingertip on an artificial skin attached to a friction board (ForceBoard™).

The properties of topical formulations can be varied a lot by just changing the ratio between water and oil. The amount of water in a topical formulation affects both the skin feel during application and the afterfeel of the formulation on the skin surface as the water evaporates. Emollients can vary in molecular and chemical structure affecting their melting point, polarity, and viscosity. Emollient type has been shown to affect skin feel, skin friction, and spreadability of the topical formulations. Furthermore, emollients can also induce occlusive effects which hydrate the skin by lowering transepidermal water loss (TEWL) (Gore et al., 2018; Greenaway, 2010; Nacht et al., 1981; Parente et al., 2005; Rawlings et al., 2004; Shai et al., 2009). Humectants are hydrophilic compounds with the capacity to retain skin water, thus maintaining hydration and minimizing water loss (Alber et al., 2013; Lodén M., 2003; Tang et al., 2015). The stability of formulations can be improved by addition of rheological modifiers that add body and thickness, and cosmetic powders are commonly added to the formulations to improve skin feel during and after application (Moussour et al., 2016; Timm et al., 2012; Wang et al., 1999). Synthetic surfactants are normally used to stabilize emulsion droplets, but they could be toxic or irritating especially to patients with sensitive skin (Berardesca et al., 2013; Lu and Moore, 2012; Veenstra et al., 2009; Wibbertmann et al., 2011). Particle-stabilized emulsions, so called Pickering emulsions, have gained a lot of interest recently, partially related to the above-mentioned formulation demands such as producing a mild surfactant-free product, and the possibility to use natural ingredients, and create new textures. But the main interest in Pickering emulsions is related to the fact that these emulsions can have high long-term stability towards coalescence and Ostwald ripening (Albert et al., 2019; Aveyard et al., 2003; Binks, 2002; Sjöö et al., 2015; Timgren et al., 2013; Venkataramani et al., 2020). While much of the work published on Pickering emulsions focuses on studying the stability and functional properties of such

systems, little attention has been given to study their sensory and cosmetic properties during application and afterfeel. In 2012, Marku et al. (Marku et al., 2012) studied the sensorial properties of Pickering emulsions stabilized with quinoa starch with a small human panel. The study reported on the impact of different oil phases but did not discuss the influence of starch particles. Marto et al. (Marto et al., 2018) later evaluated the cosmetic properties of W/O Pickering emulsions stabilized with aluminum starch octenyl succinate with a simple questionnaire. This work did not provide further understanding on the sensorial impact of starch particles. Recently, a method to study the texture and sensory properties of Pickering emulsions stabilized with inorganic particles was developed (Terescenzo et al., 2020). These authors focused on sensory analysis of Pickering emulsions stabilized with three types of solid particles, solely or in combination with a conventional surfactant, highlighting the particle effect on the sensory properties of formulations. To conclude, none of these studies have conducted a combined approach to understand and relate sensorial attributes to physical properties of starch-stabilized Pickering creams and traditional surfactant-stabilized creams.

In this paper we aim to compare, in a range of different topical creams, sensorial perception by a human panel with measurable physical properties of creams using rheology and tactile friction on porcine skin, in combination with studying effects on skin hydration. The novelty in using porcine skin for tactile friction measurements lies in allowing real-time recording of touch on skin-skin interaction between the fingertip and porcine skin. The objective is to study possible relationships between physical properties and sensorial characteristics and to elucidate the effect of different excipients on the interplay, with particular emphasis on traditional surfactant-based creams and a starch-stabilized Pickering cream. Specifically, we would like to investigate how presence of Pickering particles, surfactants, and alternative emollients affect sensorial properties during application and the afterfeel of the residual film.

## 2. Material & methods

### 2.1. Materials

Modified quinoa starch with 0.5 – 3 µm diameter granules (mean diameter = 1.7 µm), kindly provided by Speximo AB (Lund, Sweden), was used as stabilizing particles. The starch was hydrophobically modified with octenyl succinic anhydride (Timgren et al., 2013). The emollients used were medium chained triglyceride (MCT) (Miglyol 812 N, IOI Oleochemical, Hamburg, Germany), isotridecyl isononanoate (Crodamol TN, Croda, East Yorkshire, England), dimethicone (BRB DM 350, BRB International BV, Thorn, The Netherlands), hydrogenated coco-glyceride and canola oil (Akosoft 36 and Lipex Preact, AAK, Karlshamn, Sweden), jojoba oil (Natura-Tec, Fréjus, France). Liquid paraffin oil was purchased from Sigma-Aldrich (Stockholm, Sweden).

The surfactants used for surfactant-based formulations were cetearyl alcohol (Nafol 1618H, Sasol Performance Chemicals, Hamburg, Germany), PEG-100 stearate (Myrj S100), and glyceryl stearate (Cithrol GMS 40) obtained from Croda Europe (East Yorkshire, England).

Other functional excipients were Carbopol Ultrez 30 (Lubrizol, Brussels, Belgium) (also called carbomer), used as a rheological modifier and tocopheryl acetate (Dermafeel E74, Evonik Dr Straetmans, Hamburg, Germany) was used as an antioxidant. Glycerol (Sigma-Aldrich, Stockholm, Sweden) was used as a humectant and the preservatives used were phenoxyethanol and caprylyl glycol (Versatil PC, Evonik Dr Straetmans, Hamburg, Germany), propyl-4-hydroxybenzoate (Propyl paraben, Solbrol P) and methyl-4-hydroxybenzoate (Methyl paraben, Solbrol M) were provided by Lanxess GmbH (Leverkusen, Germany). Milli-Q water, 18.2 MΩ cm resistivity, was used for all samples.

## 2.2. Formulations

All the formulations studied in this work were oil-in-water (O/W) emulsions. A commercial surfactant-based pharmaceutical cream was included (Canoderm®) for comparison, purchased from a local pharmacy in Sweden. The creams prepared in the lab (batch size ~ 100 g) were a surfactant-free Pickering cream with carbomer (PC.c), a surfactant-based cream (SC.c) with carbomer as thickener and equivalent composition to PC.c, and a surfactant-based cream (SC) without a thickener and with a different emollient composition. The compositions of the cream formulations are given in Table 1.

The PC.c was prepared by mixing the water-soluble and oil-soluble excipients separately. The oil phase was heated to 60 °C until all emollients were melted, and the temperature was brought down to < 45 °C. The starch was added to the water phase below 45 °C, during mixing with a propeller stirrer (IKA RW-20, IKA, Germany), mixed for additional 5–10 min before slowly adding the oil-phase with stirring. The mixture was stirred for an additional 5–10 min before emulsification with a high shear mixer (IKA Ultra Turrax T25, IKA, Germany) at 15000 rpm for 1 min in a glass beaker.

The SC and SC.c formulations were prepared in a similar way, where the oil-phase and water-phase were mixed separately at 65–75 °C until all ingredients were dissolved, before adding the oil-phase to the water-phase while mixing with a propeller stirrer. The mixture was stirred for 5 min before emulsification with a high shear mixer at 12000 rpm for 1–2 min and allowed to cool down to room temperature during mixing.

## 2.3. Preparation of skin substrates

Fresh porcine ears were acquired from a local abattoir and stored at

– 80 °C. The ears were residuals from food preparation. No pigs were sacrificed for the purpose of this study, hence ethical permission was not required. To prepare the skin substrate, thawed pig ears were cleaned, and hair was removed with a trimmer. Full-thickness skin was excised from the inner ear using a scalpel and cut into strips (2 × 5 cm<sup>2</sup>). Finally, the excised porcine skin was wrapped in aluminum foil and stored in – 20 °C until use. Before conducting tactile friction measurements, the skin was allowed to thaw and equilibrate at room temperature at least 1 h) with a hydrated filter paper underneath. Skin from different porcine ears and position on the ear was randomized among the measurements.

## 2.4. Sensory evaluation

Sensory perception evaluation was carried out on the four moisturizing cream formulations (PC.c, SC.c, SC and Canoderm®) for perceptual attributes during application and afterfeel. Thirty-one healthy untrained volunteers (19 females, 12 men), with no history of skin disease, were recruited among students and university personnel at the Faculty of Health and Society (Malmö University) to participate as assessors in the study in accordance with ISO 8586:2012. The age ranged between 18 and 64, whilst most participants were in the age range 25–34 (67.7%). Ethical approval was received by the Swedish Ethical Review Agency (DNR 2019–05452) to conduct the study, and informed consent was obtained by all volunteers.

The evaluations were performed with assessors seated in individual booths with partition walls and homogenous artificial lightning in a dedicated room isolated from external disturbances, with temperature and humidity control in accordance with ISO 8589:2007. The study was divided into sessions of 4–6 assessors per session. Prior to each session, the assessors were asked to wash and dry their hands and were

**Table 1**  
Composition of investigated cream formulations in (wt%).

| Description                     | Functional category                                                      | Pickering cream | Surfactant-based replica of Pickering cream | Surfactant-based cream | Surfactant-based commercial cream |
|---------------------------------|--------------------------------------------------------------------------|-----------------|---------------------------------------------|------------------------|-----------------------------------|
| Cream id                        |                                                                          | PC.c            | SC.c                                        | SC                     | Canoderm® *                       |
| Water                           | Solvent                                                                  | to 100.0        | to 100.0                                    | to 100.0               | Y                                 |
| Glycerol                        |                                                                          | 5.0             | 5.0                                         | –                      | –                                 |
| Urea                            | Humectants <sup>1,2</sup>                                                | –               | –                                           | –                      | Y                                 |
| Propylene glycol                |                                                                          | –               | –                                           | –                      | Y                                 |
| Glyceryl polymethacrylate       | Film-forming <sup>1</sup>                                                | –               | –                                           | –                      | Y                                 |
| Carbomer                        | Rheology modifier                                                        | 0.10            | 0.10                                        | –                      | Y                                 |
| Modified Quinoa starch          | Stabilizing particles                                                    | 10.0            | –                                           | –                      | –                                 |
| PEG-100 stearate                |                                                                          | –               | 2.0                                         | 2.0                    | Y                                 |
| Glyceryl stearate               | Surfactants / emulsifiers <sup>1</sup>                                   | –               | 2.0                                         | 2.0                    | –                                 |
| Cetearyl alcohol                |                                                                          | –               | 2.0                                         | 2.0                    | Y                                 |
| Polysorbate 60                  |                                                                          | –               | –                                           | –                      | Y                                 |
| MCT-oil**                       |                                                                          | 12.0            | 12.0                                        | –                      | Y                                 |
| Isotridecyl Isonnanoate         |                                                                          | 4.0             | 4.0                                         | –                      | –                                 |
| Canola oil                      |                                                                          | 4.5             | 4.5                                         | –                      | Y                                 |
| Hydrogenated<br>Coco-glycerides | Emollients                                                               | 3.0             | 3.0                                         | –                      | –                                 |
| Jojoba-oil                      |                                                                          | 3.0             | 3.0                                         | –                      | –                                 |
| Solid paraffin                  |                                                                          | –               | –                                           | –                      | Y                                 |
| Liquid paraffin oil             |                                                                          | –               | –                                           | 15.0                   | –                                 |
| Dimethicone                     |                                                                          | –               | –                                           | 15.0                   | Y                                 |
| Tocopheryl acetate              | Antioxidant <sup>2</sup>                                                 | 0.50            | 0.50                                        | –                      | –                                 |
| Phenoxyethanol/Caprylyl glycol  |                                                                          | –               | –                                           | 0.70                   | –                                 |
| Ethyl paraben                   | Preservatives <sup>1</sup>                                               | –               | –                                           | –                      | Y                                 |
| Propyl paraben                  |                                                                          | 0.20            | 0.20                                        | –                      | –                                 |
| Methyl paraben                  |                                                                          | 0.20            | 0.20                                        | –                      | Y                                 |
| Lactic acid                     | Buffering, water-binding <sup>2</sup><br>/skin-conditioning <sup>1</sup> | –               | –                                           | –                      | Y                                 |
| Citric acid                     | Buffering <sup>1</sup>                                                   | –               | –                                           | –                      | Y                                 |

\* Composition according to ingredient list on product. Y = yes.

\*\* MCT = medium chain triglyceride.

<sup>1</sup> (Rowe et al., 2013).

<sup>2</sup> (Lodén, 2005).

introduced to the test procedure and the terminologies by the project leader. The introduction allowed for an automatic 20–30 min quiet acclimation period for the assessors. The assessors had been further asked to avoid caffeine beverages 3 h before the session, not to cover their forearms, and not to apply any topical formulation 12 h prior to the session in accordance with guidelines for *in vivo* measurements on skin (Berardesca et al., 2018; Otto et al., 2009; Pinnagoda et al., 1990).

Each sample was assigned a random three-digit code and presented in an identical dark glass container. The order of presentation was balanced by randomized complete block design to avoid first order and/or carry-over effects within sensory sessions. The sensory attributes were assessed on two defined circular measurement sites ( $d = 4$  cm) on each volar forearm, placed 7 cm from the wrist and elbow, and distanced 2 cm from each other. Each circle was labelled with a sticker with the formulation 3-digit code according to the randomized order. The assessors were given similar labels on the back of their fingers, as a reminder to use one finger for each measurement site to avoid carry-over effects and confusion.

*In vivo* baseline measurements of skin hydration for each measurement site were taken prior to application of 50  $\mu$ l of each cream by the project leader to the middle of the circular measurement sites using a M1000 positive displacement pipette (Microman®, Gilson, France). Once the creams were applied, assessors were allowed to start the evaluation following the instructions on a digital survey (Microsoft - Forms) on a tablet computer. The study was divided between attributes evaluated during the application phase, and attributes evaluated in the afterfeel phase when volatile compounds have evaporated (11 min after application). The sensory attributes evaluated, and instructions are summarized in Table 2. After evaluating the attributes during application, the assessors were asked to clean their fingers with facial tissues and answer a questionnaire with background information during the waiting time before the afterfeel evaluation phase. The creams were ranked for each attribute according to the rank test (ISO 8586:2006) (Meilgaard et al., 2016; Whelan, 2017), rank ordering the four formulations from 1 to 4 for the highest to lowest perception for each specific attribute. After ranking the formulations, the assessors could choose their preferred cream formulation for each studied attribute. They were also asked to choose their overall preferred formulation during the application phase and between the creams overall. By the end of the study, the assessors were also asked to describe each formulation by choosing words from a word cloud, or by using their own words.

## 2.5. Skin hydration measurements

Skin hydration was measured *in vivo* during the sensory perception study before the application of creams, and upon completion of the study by electrical conductance by means of a hydration probe (Dermalab, Cortex Technology, Denmark) at circular measurement sites on the volar forearms. The instrument measures skin conductance at a single frequency, 300 kHz, which can be related to the water content of *stratum corneum* on an arbitrary scale (Morin et al., 2020). The results are given as the relative change (%) in  $\mu$ S (microSiemens) on each measurement site to evaluate the effect of the formulations on skin hydration.

*Ex vivo* skin hydration measurements were performed during tactile friction measurements, using a Corneometer (CM825, Courage Khazaka Electronic GmbH). The principles for the instrument are based on capacitance measurement and it was used to measure the skin hydration of the finger and the excised skin, prior to each experiment. A second measurement was performed after each experiment on the excised skin. The results are given as the relative change (%) in arbitrary units (a.u) on excised skin to evaluate the effect of skin hydration on friction results.

**Table 2**

The attributes evaluated using a ranking test, given in the order they were evaluated in the study with definitions and instructions as specified in the survey. 50  $\mu$ l of each formulation was applied on circular sites on the volar forearms (two sites per forearm).

| Attributes, Application phase | Definition                                                               | Instructions                                                                                                                                                                  |
|-------------------------------|--------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Moistness                     | The amount of liquid (wetness) which can be perceived during application | Spread the sample droplet within the marked circle in a circular motion with 1 finger. (1 circle/second). After 3 circulations, evaluate how moist the samples are.           |
| Spreadability                 | The ease to spread the sample over a larger skin area                    | Continue distributing the sample on the skin in circular motion (1 circle/second). After another 2 circulations (total 5), evaluate how spreadable the samples are.           |
| Thickness                     | The amount of sample-film between the finger and the skin                | Continue distributing the sample in a circular motion (1 circle/second). After another 7 circulations (total 12), evaluate the thickness of the samples.                      |
| Absorption                    | How fast the sample absorbs into your skin                               | Continue distributing the sample in a circular motion (1 circle/second). Max 18 additional circulations (total max 30) Evaluate how fast the sample is absorbed by your skin. |
| Attributes, Afterfeel phase   | Definition                                                               | Instructions                                                                                                                                                                  |
| Stickiness                    | How sticky/gluey the skin feels                                          | Press your finger withing the marked circle and lift it to evaluate the stickiness. You may repeat it 3 times. Evaluate how Sticky the skin area feels.                       |
| Residual Coating              | The amount of remaining product on the skin area                         | Use your finger to slide softly over the skin in the marked circles and evaluate how much product that remains.                                                               |
| Greasiness                    | Amount of oil/fat which can be perceived on the skin surface             | Slide your finger within the marked circles and evaluate how greasy/oily the skin area feels.                                                                                 |
| Slipperiness                  | How slippery the skin feels                                              | Let your finger slide over the skin in the marked circles and evaluate how slippery the area feels.                                                                           |
| Softness                      | How soft and smooth the skin feels                                       | Let the finger slide slowly in the marked circles and evaluate how soft the area feels.                                                                                       |

## 2.6. Rheological studies

Rheological measurements were performed using a stress-controlled rheometer, Bohlin CVO100 (Malvern Instruments, UK) equipped with a parallel plate with a diameter of 25 mm and a gap size of 0.50 mm. The formulations were applied to the bottom plate with a stainless-steel spatula and analyzed at 32 °C. Continuous flow tests (hysteresis loop tests) of the formulations were carried out with shear rate ramp, by increasing the shear rate from 0.25 to 200  $s^{-1}$ , hold time of 0.1 s, and decreasing to 0.25  $s^{-1}$ . The duration of each step was 10 s. The steady shear viscosity and hysteresis loop area values were calculated from the obtained curves (shear stress vs shear rate) using the Bohlin Rheometer



Software. Dynamic (oscillatory) tests were performed to determine the linear viscoelastic region (LVR) and yield stress of the samples, at constant frequency of 1 Hz and an amplitude sweep ramp from 0.24 to 1000 Pa. A frequency sweep was performed in the linear region from 0.1 to 1000 Hz at 0.3 Pa constant shear stress. The storage ( $G'$ ) and loss ( $G''$ ) moduli phase angle ( $\delta$ ) was measured. The yield stress was determined from the onset value of the modulus curves in a double logarithmic plot of the storage modulus as a function of oscillation stress (Dinkgreve et al., 2016; Walls et al., 2003).

## 2.7. Tactile friction measurements

Tactile friction measurements were performed using a ForceBoard™ (Industrial Dynamics Sweden AB, Järfälla, Sweden), following the method described by Skedung et al. (Skedung et al., 2016) with minor adjustments. The instrument is equipped with both a horizontal and one tangential load cell, consisting of strain gauges in a Wheatstone bridge configuration. A mechanical load results in voltage changes that are proportional to the applied load. A temperature-controlled plate was placed on top of the ForceBoard™ (32 °C) and excised porcine skin (2x5 cm<sup>2</sup>) was mounted on the heated plate with hydrated filter paper underneath. The friction force ( $F$ ) and applied load ( $L$ ) were continuously recorded as a finger interrogated the model skin surface by moving the index finger back and forth, and the friction coefficients ( $\mu$ ) were calculated as a ratio of the friction force and load according to:

$$\mu = \frac{F}{L} \quad (1)$$

Approximately 4–5 mg cm<sup>-2</sup> of the topical formulations (PC.c, SC, and Canoderm®) were applied to the skin substrate. For each experiment the index finger, inclined 30°, was stroked forward and back 10 times to spread 50  $\mu$ l (20–25 mg) of applied formulation over the sample area (4–5 cm<sup>2</sup>). A control measurement on untreated skin substrate with 10 S was performed prior to each measurement as control for changes in skin and finger. The friction was measured for 30 s during application of the formulation at the time points 2.5 min, 5.5 min, and 11 min after application. All experiments were performed in controlled ambient conditions (temperature = 21 °C  $\pm$  0.3 °C, relative humidity = 49%  $\pm$  6%) by the same experimenter. The method was validated by comparison with another experimenter and an artificial skin substrate (unpublished results). The friction force and the applied load were recorded with a sampling rate of 100 Hz and the applied load was maintained around 0.5 N. The results are given as average friction coefficient values normalized against corresponding values for the untreated skin substrates to control for biological variation between individual skin substrates. The results for untreated skin substrates show a small reduction in normalized friction coefficient over the measurement period of 11 min, tentatively resulting from water evaporation with time (unpublished results).

## 2.8. Statistical analysis

Whenever applicable, results were expressed as averages  $\pm$  standard deviation, and differences between cream formulations were determined by one-way ANOVA with Tukey's HSD test. Statistical outliers were excluded based on two-sided Grubbs' test ( $p < 0.05$ ).

A Friedman test of difference was used to determine whether participants had a significant different rank ordered preference for the four cream formulations ( $p < 0.05$ ). The Friedman test is the non-parametric alternative to the one-way ANOVA and shows whether significant differences exist between two or more samples that are ranked by all panelists. It is a very sensitive test to find a pattern of consistent rank order (Lawless and Heymann, 2010). Whenever there was a significant difference, pairwise comparison was performed using Fisher's least-significant-difference (LSD) test to determine which of the cream formulations was significantly different ( $p < 0.05$ ).

Multivariate analysis was carried out using Matlab (v. R2020b). Linear partial least squares regression analysis (PLS) was used to visualize the relationships between the studied creams, the rank sums of the sensory attributes and the mean data for the physical measurements.

## 3. Results

### 3.1. Participants' background

Questionnaire results provided background information about the participants in the sensory perception study (Appendix A, [supplementary information](#)). Most of the participants were females (61%), and a large number of the participants were in the age range 25–34 years old (67%). Almost half of the participants (48%) use hand cream on a daily basis, while the rest use it only on a weekly basis (19%), seasonally (19%), or never (4%).

Most of the participants preferred non-greasy (>38%) and soft, cushioning (>38%) moisturizing creams. Some participants (15%) preferred their moisturizing creams to be thin as well as non-greasy/soft, cushioning. Upon ranking the most important criteria when purchasing a new moisturizing cream, the top three criteria most important for the participants were the moisturizing effect, the afterfeel sensation, and texture. Based on this information we can conclude that the afterfeel sensation of the applied formulation is important for the participants in this study, and that a cream with a moisturizing effect and a non-greasy and soft afterfeel is more likely to be preferred.

### 3.2. Sensory perception evaluation

The results of the sensory study (Table 3) show statistically significant differences ( $p < 0.05$ ) in participant's ranked preference between the four cream formulations for all sensory attributes except thickness and absorption. The rank sums of the cream formulations for each sensorial attribute are illustrated in a radar chart in Fig. 1A.

#### 3.2.1. Perception during the application phase

The attributes evaluated during the application phase are relevant to the cosmetic appeal of creams, and formulations ranked high for these attributes are generally considered as cosmetically appealing for consumers. Moistness, and absorption are related to the sensation of the skin being moisturized and hydrated, while thickness and spreadability are related to a pleasant feeling and the ease of application. During the application of the topical creams to the skin, the formulations need to spread easily without feeling too greasy or sticky (Kwak et al., 2015; Vergilio et al., 2021). Formulations ranked high for these attributes can be considered as cosmetically appealing for consumers.

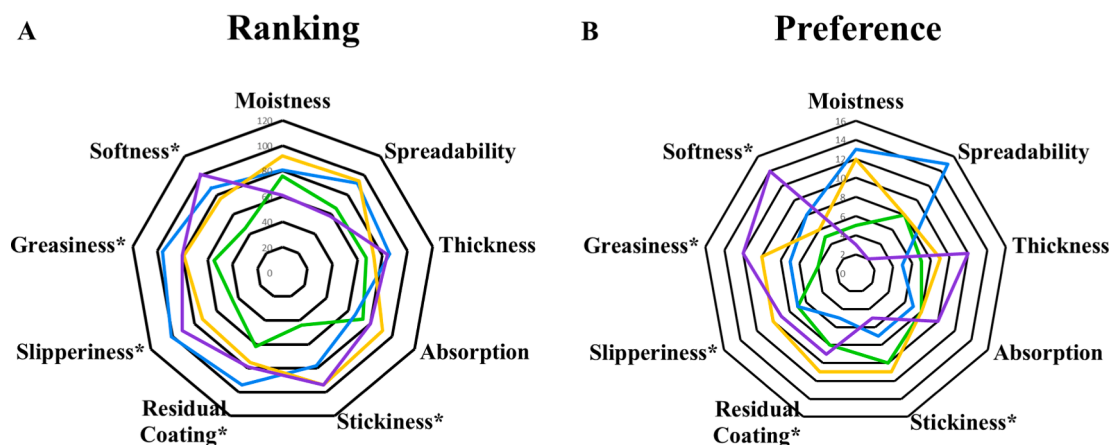
During the application phase, Canoderm® was perceived as less

**Table 3**

For each sensory attribute, Chi-square values ( $\chi^2$ ), and p-values from Friedman's test and rank sums for the cream samples with multiple comparisons using Fisher's least-significant-difference (LSD,  $p < 0.05$ ). High rank sums correspond to high level of perception for each sensory attribute.

| Attributes       | $\chi^2$ | p-value | PC.c <sup>1</sup> | SC.c <sup>2</sup> | SC3              | Canoderm®        |
|------------------|----------|---------|-------------------|-------------------|------------------|------------------|
| Moistness        | 9.62     | 0.022*  | 76 <sup>ab</sup>  | 81 <sup>b</sup>   | 92 <sup>b</sup>  | 61 <sup>a</sup>  |
| Spreadability    | 19.26    | <0.001* | 66 <sup>a</sup>   | 92 <sup>b</sup>   | 94 <sup>b</sup>  | 58 <sup>a</sup>  |
| Thickness        | 4.74     | 0.192   | 67 <sup>a</sup>   | 86 <sup>a</sup>   | 73 <sup>a</sup>  | 84 <sup>a</sup>  |
| Absorption       | 6.60     | 0.086   | 73 <sup>a</sup>   | 66 <sup>a</sup>   | 91 <sup>a</sup>  | 80 <sup>a</sup>  |
| Stickiness       | 32.26    | <0.001* | 44 <sup>a</sup>   | 78 <sup>b</sup>   | 94 <sup>b</sup>  | 94 <sup>b</sup>  |
| Residual Coating | 10.08    | 0.017*  | 62 <sup>a</sup>   | 94 <sup>b</sup>   | 75 <sup>ab</sup> | 77 <sup>ab</sup> |
| Greasiness       | 16.59    | <0.001* | 55 <sup>a</sup>   | 96 <sup>b</sup>   | 79 <sup>b</sup>  | 80 <sup>b</sup>  |
| Slipperiness     | 35.05    | <0.001* | 45 <sup>a</sup>   | 101 <sup>c</sup>  | 73 <sup>b</sup>  | 91 <sup>c</sup>  |
| Softness         | 31.68    | <0.001* | 46 <sup>a</sup>   | 87 <sup>bc</sup>  | 76 <sup>b</sup>  | 101 <sup>c</sup> |

\* for significant results,  $p < 0.05$ . Creams sharing the same significance group letter (a,b, c) show no difference in ranked perception. <sup>1</sup>PC.c = Pickering cream with carbomer. <sup>2</sup>SC.c = Surfactant-based cream with carbomer. <sup>3</sup>SC = Surfactant-based cream.



**Fig. 1.** Radar charts of rank sums (A) and preference score (B) of each cream formulation for the studied sensory attributes. Sensory attributes studied during the afterfeel phase are star marked. A: Rank sums of each cream formulation for the studied attributes. A higher value of rank sums for a certain sensorial attribute means a stronger perceived intensity for this attribute for a given formulation (e.g., most soft). B: Preference score of each cream formulation for the studied attributes, showing which cream was most preferred for a specific attribute. The formulations were Pickering cream (PC.c) in green, surfactant-based cream replica of Pickering cream (SC.c) in blue, surfactant-based cream (SC) in yellow, and surfactant-based commercial cream (Canoderm®) in purple.

moist than SC.c and SC ( $p < 0.05$ ), and together with PC.c it was perceived as less spreadable than SC.c and SC ( $p < 0.05$ ). The highest rank sums overall for the sensory attributes during the application phase were given to SC for all sensory attributes except thickness, where SC.c and Canoderm® were higher. Thus, we can conclude that SC was perceived as the moistest, the easiest to spread, and the cream to absorb fastest by the skin during the application phase.

### 3.2.2. Perception during the afterfeel phase

According to the human panel in this study, the afterfeel sensation is an important criterion when purchasing a cream. Most participants preferred a non-greasy and/or soft afterfeel. Thus, a high rank sum value for softness and a low value of rank sums for greasiness can be interpreted as being cosmetically appealing for this specific panel. In general, some of the attributes related to the afterfeel phase are commonly considered unfavorable; such as stickiness, greasiness and slipperiness (Kwak et al., 2015; Lodén M, 2003; Nacht et al., 1981; Savary et al., 2019), while softness can be seen as a favorable attribute, e.g., a powdery afterfeel (Moussour et al., 2016; Timm et al., 2012). Low values of rank sums for stickiness, and slipperiness can also be considered as cosmetically appealing.

In the afterfeel phase, 11 min after the formulations were applied, the participants' ranked preferences showed that the lowest rank sums ( $p < 0.05$ ) were given to PC.c for all afterfeel sensory attributes. The afterfeel of PC.c was thus perceived as the cream with least stickiness, greasiness, residual coating, slipperiness but also the least soft. Canoderm®, SC and SC.c were perceived as sticky and greasy in afterfeel, with high rank sums for stickiness and greasiness. The highest amount of residual coating was perceived for SC.c in the afterfeel phase, significantly different from PC.c ( $p < 0.05$ ). Furthermore, the residual films of SC.c and Canoderm® were perceived as most slippery on skin, significantly higher than PC.c and SC ( $p < 0.05$ ) separately, and the cream that was perceived to leave the softest ( $p < 0.05$ ) skin sensation in the end was Canoderm®.

### 3.2.3. Preference choice

The assessors were also asked to choose the cream they preferred the most for each attribute during application and afterfeel phases, Fig. 1B. During the application phase, SC.c, was preferred the most for moistness and spreadability attributes while Canoderm® was the most preferred cream for thickness and absorption attributes. For the afterfeel attributes, Canoderm® was the most preferred for softness and slipperiness while SC was most preferred for stickiness, residual coating, and

greasiness. SC was the overall most preferred cream for the application attributes. When asked for the most preferred cream overall, without thinking of a certain attribute, Canoderm® was the most preferred cream. Canoderm® is a fully developed product and marketed as an efficient moisturizer, in comparison to the prototypes prepared in the lab, and this difference was perceived by the assessors when they only needed to compare the overall performance between all four creams.

The preference results differed to some extent from the results by ranking. This highlights how the participants are in different state of mind when asked to compare and rank the creams based on level of perception for each attribute, and when asked to choose the cream they preferred the most. In the first case, they are in an analytical frame of mind when comparing and ranking, while they are looking at the cream as a whole and judging based on their own preferences when asked to choose the most preferred (Lawless and Heymann, 2010). Preference tests would require a larger number of assessors or trained panelists and preference results were only considered as supplement to the rank test.

### 3.2.4. Participants' descriptive words

At the end of the sensory study, the participants were asked to describe each cream formulation with their own words. A word cloud animation was provided as assistance. The words used to describe each cream formulation are illustrated in Fig. 2, with colors and font size highlighting how frequent each word was used. The words that were used the most to describe PC.c were "grainy" and "easily absorbed", while SC.c was described as "greasy" and "oily". Canoderm® was described as "smooth", "soft", and "greasy" and SC was described as "soft", "natural" and "oily".

The most common words used to describe all three surfactant-based creams SC, SC.c and Canoderm® were "soft", "greasy", "oily", and "smooth". These words were either not used at all or less frequently used to describe the surfactant-free PC.c. Soft and smooth relates the sensation of the formulation film, while greasy and oily relates to the amount of residual oil film perceived after application. The words that were frequently and solely used to describe PC.c besides "grainy" and "easily absorbed" were "powdery feeling", "dry", "dry residue", "rough", "matte", and "bad quality". Most of these words indicate that the participants could feel the presence of starch particles either as a result of dry afterfeel or as powdery, rough, matte feeling and that some of these sensations were associated with "bad quality" for some of the assessors. Surfactant-based creams can be perceived as soft and smooth, while resulting in a greasy and oily afterfeel. In contrast, a surfactant-free Pickering cream is perceived as grainy but with a dry, powdery



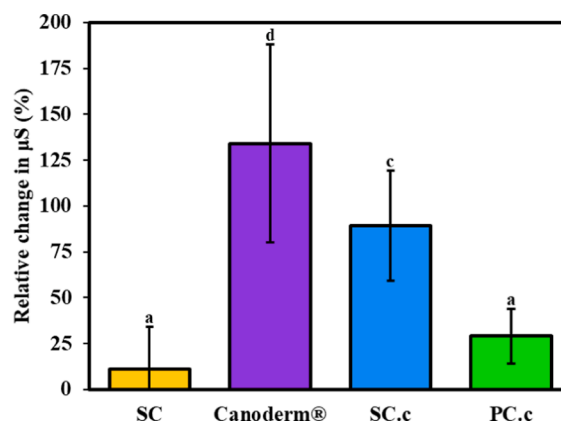
**Fig. 2.** Word clouds of the words used to describe each cream formulation. Difference in frequency of used word is highlighted by colors and font size (larger size for higher frequency).

afterfeel. These results highlight the impact of the presence of starch particles in the residual film on the perception of greasiness, stickiness, and oiliness. However, the large diversity of words used to describe each cream indicate that it was difficult to describe the overall skin feel of each cream. It is also important to keep in mind that the panel consisted of untrained subjects and the individual expectations and preferences may impact how the creams are perceived. We can conclude that there is a difference in how participants perceived traditional surfactant-based creams and the surfactant-free Pickering cream.

### 3.2.5. Hydration measurements *in vivo*

The results of the hydration measurement (Fig. 3) of the skin before and after the application of creams can help distinguish the degree of hydration to the skin after application of the creams. The relative change (%) in  $\mu\text{S}$  was significantly highest for Canoderm® ( $p < 0.05$ ), followed by SC.c. Even though SC.c and PC.c have the same composition of excipients and differ only by the addition of starch particles instead of surfactants, the relative change in skin hydration was significantly higher for SC.c ( $p < 0.05$ ). The fatty and hydrophobic composition of the surfactants can contribute to an occluding effect giving rise to a higher value in skin hydration, while the presence of starch particles may alter the occluding properties of the residual oil film.

The difference between the surfactant-based creams could be related to difference in oil composition and presence of humectants. Furthermore, Canoderm® is marketed as an efficient moisturizer with 5% urea as well as propylene glycol as humectants, compared to glycerol which is present in PC.c and SC.c. Urea has been shown to be an efficient humectant to increase skin hydration (Albèr et al., 2013), resulting in lower TEWL in comparison to glycerol (Lodén et al., 2001). Hence, addition and choice of humectants together with occlusive emollients significantly affect the moisturizing properties of a cream.



**Fig. 3.** Relative change in conductance,  $\mu\text{S}$ , after application of cream formulations *in vivo*. Results are expressed as average ( $n = 30$ ) and error presented as standard deviation, and differences between cream formulations were determined by one-way ANOVA with Tukey's HSD test. Creams with different group letter (a, b, c, d) are significantly different ( $p < 0.05$ ) in relative change in hydration.

### 3.3. Rheological studies

#### 3.3.1. Flow curves

The flow properties of a cream can tell us how easy it is to apply and spread a cream on the skin. It has been suggested that a skin cream should have low viscosity at high shear to be easy to apply, and high viscosity at low shear so it does not spill out of the container (Greenaway, 2010). The flow behavior of the studied creams (Fig. 4) show that all creams exhibited shear-thinning and thixotropic behavior. It can be



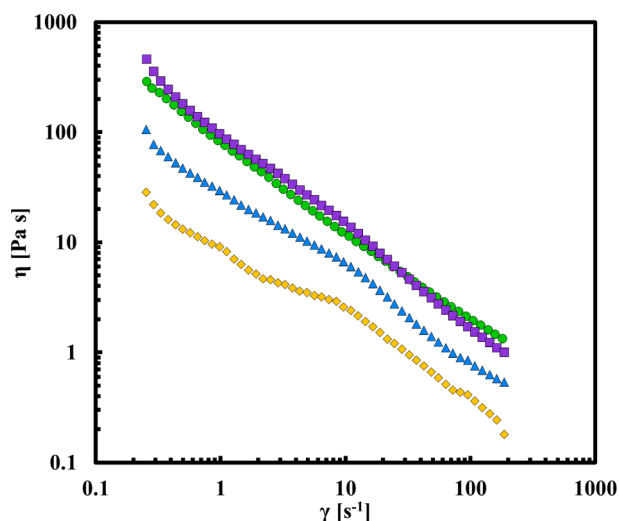


Fig. 4. Shear-thinning behaviour for all formulations. Pickering cream (PC.c), green circles, surfactant-based Pickering cream replica (SC.c), blue triangles, surfactant-based cream (SC), yellow diamonds, and surfactant-based commercial cream (Canoderm®), purple squares.

noted that steady shear viscosity (at low shear rate) was highest for Canoderm® and PC.c, followed by SC.c and SC. The creams with higher viscosity all had in common the presence of a structural thickener (carbomer). The presence of starch in the PC.c could explain the higher initial viscosity of PC.c in comparison to SC.c. SC exhibited the lowest shear viscosity and differed the most from the other creams.

### 3.3.2. Oscillation measurements

Yield stress is a measure of the stress required to induce flow in a product and can be related to the interparticle structure. Results from oscillation amplitude sweep measurements are shown in Table 4.

All formulations showed elastic behavior at low stress, indicated by higher  $G'$  values than  $G''$  values, and displayed a LVR up to approximately 3 Pa. Upon further increase of the shear stress, the  $G'$  values drop for some formulations and the viscous flow effects is apparent with  $G''$  leading to cross over. Yield stress results were determined from the onset of the curve of  $G'$  (Dinkgreve et al., 2016) (Figure A1, Appendix A) and summarized in Table 3. The highest yield stress recorded was 129.7 Pa for Canoderm®. PC.c exhibited lower yield stress (77.8 Pa) than Canoderm® but higher than SC.c (33.2 Pa). The difference between PC.c and SC.c can be attributed to the difference in using surfactants or starch particles as emulsion stabilizers and the difference in microstructure. The lower yield stress of SC.c indicates increased breakdown of the structure in comparison to PC.c. The only cream which did not include any structural thickener was SC, and it exhibited the lowest yield stress values (<16.8 Pa).

Carbopol as a structural thickener is known to enhance stability, and add body to skin cream samples (Epstein, 2009; Greenaway, 2010; Kwak

et al., 2015). Therefore, it is not surprising that formulations including carbopol displayed higher yield stress and viscosity. However, differences beyond the addition of carbopol could relate to the amount or type of oil used and the microstructure of the oil droplets. According to Brummer et al. (Brummer and Godersky, 1999) the onset of flow for creams is above a critical shear stress of 10 Pa, whereas lotions begin to flow at lower yield stresses < 10 Pa. The four main formulations investigated here all displayed yield stress values above 10 Pa indicating that they are suitable as topical creams.

A frequency sweep was performed for all formulation at a value of shear stress within the LVR at 0.3 Pa (Table 4). The values of  $G'$  were higher than  $G''$  ( $\tan \delta < 1$ ) for all the formulations in a wide frequency range (up to 10 Hz), indicating elastic behavior and that all samples are in a gel (or solid) state. Above 10–29 Hz, the  $G''$  crosses over and becomes higher than  $G'$  ( $\tan \delta > 1$ ) showing that viscous behavior starts to dominate, and the samples enter into a liquid state. The values of  $\tan \delta$  ( $G'/G''$ ) were lowest for PC.c, which suggest that it is the most elastic sample with sturdier internal structure.

### 3.4. Tactile friction

During initial application of a cream, when the film formed between skin and finger is rather thick, the friction is highly affected by the viscosity of the formulation, and the friction properties are related to the perceived slipperiness of the product (Guest et al., 2013; Skedung et al., 2016; Tang et al., 2015). After spreading of the formulation, as the water and other volatile components evaporate, the effect of the product on the skin can be detected. For example, hydrated and soft skin gives rise to a higher friction, while a large amount of non-adsorbed oil residue on the skin may give rise to lower friction (Nacht et al., 1981; Skedung et al., 2016; Tang et al., 2015).

The average normalized friction coefficients during application on skin, and 2.5, 5.5 and 11 min after application of studied cream formulations are shown in Fig. 5. Upon application (0 min), there is an initial drop in friction of the skin due to spreading for all the creams in comparison to the untreated skin samples. Once the thickness of the formulation film is decreased and subsequent changes of the residual film occur, including the evaporation of volatile compounds, the normalized friction is increased for all formulations. PC.c shows a pronounced increase in friction after 2.5 min, and by the end of the experiment (11 min), the friction of PC.c is still higher than untreated skin. Canoderm®, SC.c and SC displayed a minor gradual increase in friction over the measuring time. After 11 min, Canoderm® reaches similar friction levels as untreated skin, while the friction for SC.c and SC ( $SC.c > SC$ ) is lower than that of untreated skin. The difference in friction between Pickering-stabilized (PC.c) and surfactant-stabilized creams (SC.c, SC, and Canoderm®) can be related to the use of starch particles instead of surfactants. The residual film in Canoderm®, SC.c and SC contains oils and surfactant residues which could contribute to more slippery and greasy tactile properties reducing the friction. As for PC.c, the residual film contains mainly oils and starch particles, where the presence of starch particles may contribute to higher tactile friction

Table 4

Rheological parameters of investigated formulations.  $G'$ ,  $G''$ , and phase angle values were obtained at 1 Hz. Yield stress was obtained from the onset of the curve of  $G'$ . Steady state viscosities and hysteresis loop areas were obtained from flow tests. Values are given as mean  $\pm$  SD.

| Creams            | Temp. (°C) | Hysteresis Loop area (Pa s <sup>-1</sup> ) | Steady shear viscosity (Pa s) | $G'$ (Pa)       | $G''$ (Pa)      | Tan $\delta$    | Yield Stress (Pa) |
|-------------------|------------|--------------------------------------------|-------------------------------|-----------------|-----------------|-----------------|-------------------|
| Canoderm®         | 32         | 4913 $\pm$ 423                             | 460.8 $\pm$ 68.6              | 6181 $\pm$ 1276 | 3287 $\pm$ 1534 | 0.51 $\pm$ 0.1  | 129.7             |
| PC.c <sup>1</sup> | 32         | 2846 $\pm$ 417                             | 288.7 $\pm$ 50.3              | 871 $\pm$ 178   | 69.2 $\pm$ 7.5  | 0.08 $\pm$ 0.02 | 77.8              |
| SC.c <sup>2</sup> | 32         | 3171 $\pm$ 37                              | 105.4 $\pm$ 20.3              | 1277 $\pm$ 115  | 359 $\pm$ 86    | 0.28 $\pm$ 0.04 | 33.2              |
| SC <sup>3</sup>   | 32         | 1875 $\pm$ 15                              | 28.5 $\pm$ 5.9                | 619 $\pm$ 201   | 229 $\pm$ 71.1  | 0.37 $\pm$ 0.01 | 16.8              |

<sup>1</sup> PC.c = Pickering cream with carbomer. <sup>2</sup>SC.c = Surfactant-based cream with carbomer. <sup>3</sup>SC = Surfactant-based cream.



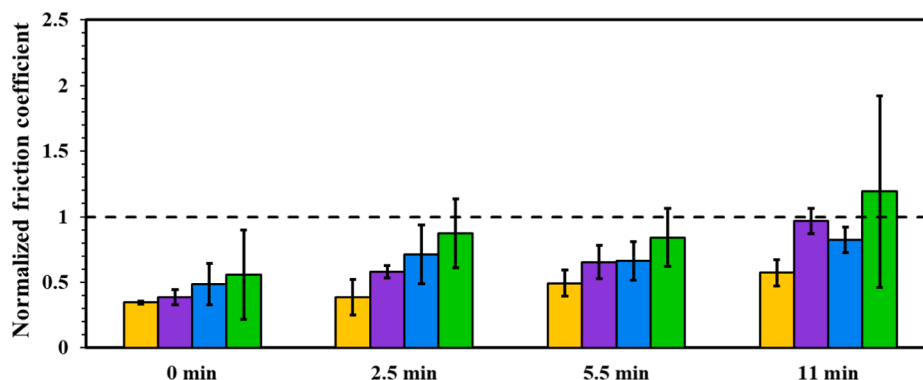


Fig. 5. Tactile friction results on excised skin at 32 °C of the studied formulations. Surfactant-based cream (SC) in yellow, surfactant-based commercial cream Canoderm® in purple, surfactant-based cream with carbomer (SC.c) in blue, and Pickering cream (PC.c) in green. One-way ANOVA showed that the SC and PC difference at 2.5 min and 5.5 min is statistically significant ( $p < 0.10$ ).

and counteract the reduction in friction caused by the residual oil. Nacht et al. (Nacht et al., 1981) concluded that greasy products have been found to induce an initial decrease in friction coefficient, while products that increase friction coefficients would be perceived as non-greasy. Thus, high friction inducing PC.c can be considered as non-greasy, while surfactant-based creams with lower friction can be perceived as greasy. Timm et al. showed that adding particles to an aqueous suspension lowered friction and friction lowering was particle size dependent (Timm et al., 2012). However, no comparisons were made with emollients or particle containing creams. It is thus not farfetched to assume that, once water evaporates from the formulation, the friction would differ between a dry particle residue after an aqueous suspension, and a residue of particles and oil mixed after an emollient suspension or a cream containing particles. Furthermore, the microstructure of the residual film may also differ for particles dispersed in a surfactant-based cream, or particles used as emulsifiers surrounding the oil droplets (Pickering emulsions). Difference among surfactant-based creams (SC.c, SC and Canoderm®) may be attributed to the different properties of the residual film due to different emollient and surfactant composition, as well as presence of thickeners.

### 3.5. Hydration measurements *ex vivo*

The relative change (%) in skin hydration (a.u) of excised skin before and after the experiment for the different treatments is shown in Fig. 6. For untreated skin samples, a reduction in skin hydration (up to 27%) was noted for the experiment period of 11 min. Thus, the water loss from the hydrated excised skin due to evaporation was high. There was tendency for relative increase in hydration 11 min after application

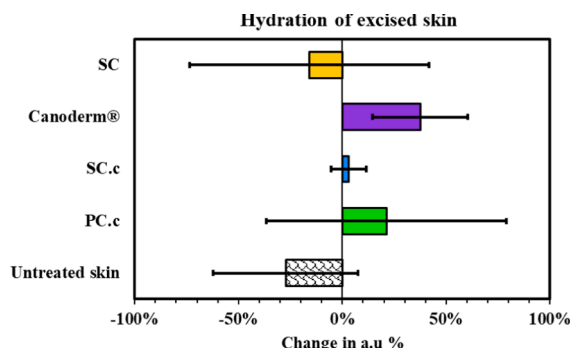


Fig. 6. Change in a.u % after application of formulations on excised skin (11 min). Surfactant-based cream (SC), yellow bar, commercial cream Canoderm®, purple bar, surfactant-based cream with carbomer (SC.c), blue bar, Pickering cream (PC.c), green bar, and untreated skin substrates, textured bar. Errors are presented as standard deviations.

observed for all creams except SC, but no significant differences in hydration were found among the creams and untreated skin. The increase in hydration was highest for Canoderm® followed by PC.c and SC.c. Canoderm® comprises humectants such as urea and propylene glycol which may have contributed to the higher skin hydration in combination with occlusive effects of the emollient composition. PC.c and SC.c both comprised glycerol as humectant, and although starch particles in PC.c may alter the properties of the occlusive film, the skin hydration for PC.c was still higher than SC.c. SC did not include any humectants and contained different emollient composition which may have contributed to the lower skin hydration. Thus, humectants when combined with an appropriate occlusive emollient composition had a larger impact on skin hydration results. The higher skin hydration levels of Canoderm® may explain the difference in friction results between Canoderm®, SC.c and SC, since hydrated skin increases friction. Canoderm® was also the cream that increased the participants skin hydration the most *in vivo*.

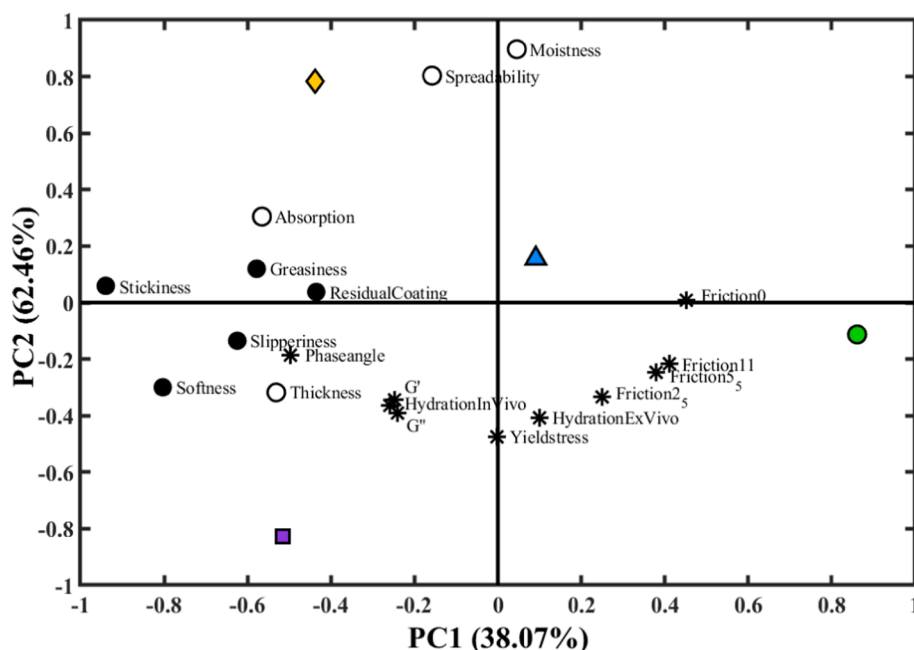
## 4. Discussion

### 4.1. Relationship between sensory and physical data and creams

In this study we have conducted a study comprising both sensory properties and physical chemical properties of a Pickering-stabilized cream and three surfactant-stabilized creams. Sensory properties are always the key attribute from a consumer perspective, but consumer panels are time consuming and thus not a fast tool during the development phase. Consequently, there is a need to link the sensory properties to easily measurable physical properties.

We conducted partial least square (PLS) mainly as a visual tool (due to limited numbers of studied creams) to study the relationships between physical data, sensory data, and the studied creams. Sensory data comprised rank sums of the creams for each sensorial attribute evaluated by thirty-one assessors, and the physical data comprised average data of replicates ( $n = 3-4$ ) for each cream and physical parameter. PLS showed that the main data variance, 62.46%, was explained by the first two principal components. The first principal component (PC1) explained 38.07% of the data variance, while the second principal component (PC2) accounted for 24.39% of the variance. A PLS plot combining the sensory data, physical data, and the studied creams (Fig. 7) suggests that PC1 is associated with afterfeel attributes and physical data, since they are generally distributed along the x-axis. Tactile friction data and *ex vivo* hydration data are positioned on the positive side of the x-axis and rheological and *in vivo* hydration data are, in general, on the negative side of the x-axis. PC2 seems to be associated with sensory attributes related to the application phase, due to their apparent distribution along the y-axis. In general, it seems as if PC2 shows differences between moist and thin creams and fatty and thick creams.

The four studied creams were distributed quite well in both



**Fig. 7.** Partial least square (PLS) visualizing the relationships between sensory data (circles), physical data (black stars) and studied creams. Sensory data are shown as attributes studied during the application phases (white circles) and during the afterfeel phase (black circles). The studied creams are Pickering-stabilized PC.c (green circle) and surfactant-stabilized creams SC (yellow diamond), SC.c (blue triangle), and Canoderm® (purple square).

dimensions of the PLS, indicating that each cream appears to have unique characteristics in their perceived attributes. Pickering-stabilized PC.c is negatively associated with most afterfeel attributes (greasiness, residual coating, and stickiness) suggesting it was perceived to have less residual coating and to be less greasy and sticky than the other creams. It was also the cream that is positively associated with tactile friction data. Canoderm®, appears to be positively associated with thickness, and negatively associated with moistness. Furthermore, Canoderm® seems to be positively associated with rheological data and hydration data and differed most from the other three creams in these aspects. SC is influenced slightly more by application attributes than afterfeel attributes and is associated with spreadability since it was ranked high for spreadability and perceived as easy to spread during application. SC is however negatively associated with friction data, indicating that it has properties that result in low friction values. The PLS suggests that SC.c had rather weak associations with the various variables, suggesting ‘middle-of-the-road’ physical measures and sensorial responses.

The surfactant-based creams appear to be separated from each other along the y-axis suggesting slightly different characteristics between the creams and mainly associated with hydration and rheological results. For instance, differences between a moist and thin cream and a thick and fatty cream can be visualized along the y-axis. On the other hand, the relationship between surfactant-based and surfactant-free creams was mainly explained by the positioning along the x-axis. Furthermore, strong differences in afterfeel perception between the surfactant-based creams due to type of emollient used could to some extent be visualized by their position along the x-axis. Thus, the differences in composition due to oil:water ratio, type of emollient used, and presence of thickeners and humectants could mainly be explained by their position along the y-axis, and partly by their position on the x-axis and can be studied by means of hydration measurements and rheological studies.

#### 4.2. Relationship between sensory and physical data

Tactile friction results and afterfeel attributes are generally negatively associated with each other, while rheological and hydration data seems to be related to some afterfeel attributes. The PLS further suggests that there seems to be negative associations between absorption and

tactile friction data in general. Spreadability seems to be negatively associated with friction results at 2.5 min, suggesting that a cream with a residual film that remains spreadable after 2.5 min is likely to have low friction values. Furthermore, spreadability seems to be negatively associated with the yield stress, as a cream with a high yield stress value is more likely to be difficult to spread. Negative associations were also found for moistness and G', G'', viscosity, and *in vivo* hydration data. Moistness was defined as the amount of liquid or degree of wetness perceived during application. Thus, it is logical to imagine that a thin cream with low viscosity is perceived as more moist by the participants. However, skin hydration measurements focus on evaluating water content of the stratum corneum by means of capacitance and conductance, and are not appropriate methods to measure moistness on skin (Berardesca et al., 2018). Thus, we cannot suspect any logical causality between moistness and hydration results. The PLS further shows that thickness and softness were positively associated with each other and associated with viscosity results.

Stickiness could not be related to rheological and tactile friction data. This can be due to that the sensory perception of stickiness is evaluated by tapping the skin with the fingertip where the movement is different from oscillating movement in rheology and the stroking movement in tactile friction data. Other studies have found correlation between compression tests and stickiness (Lee et al., 2021; Savary et al., 2019).

The present results, as visualized by PLS, suggest that tactile friction experiments could be used to determine sensorial attributes related to afterfeel, and rheological experiments could be used to determine attributes relating to the application phase such as thickness, and spreadability. These findings appear to be in line with results reported by Savary et al. (Savary et al., 2019) and Lee et al. (Lee et al., 2021).

#### 4.3. Impact of excipients on sensorial and physical properties

The results show that the functional excipients used had an impact on physical properties that could be related to sensorial properties. Humectants were the only exception since the impact of which could only be measured instrumentally, and the moisturizing effect was not evaluated by perception during the evaluation period. Moreover, although humectants can give a moisturizing effect that can change the

aesthetics of the skin after long-term use, it is unlikely that this change can be perceived short after one single application.

The presence of a rheological thickener contributed to the viscosity, yield stress and the storage stability of creams. Consequently, the viscosity and yield stress were mainly related to the initial application and spreading of the creams. The two creams with lower viscosity and yield stress, SC and SC.c, were perceived as easiest to spread. However, SC did not contain any thickener and differed from SC.c in the emollient composition. Furthermore, SC was also perceived as the moistest and quickest cream to be absorbed during application. Thus, the combination of thickener and emollients is important to consider when designing a formulation with specific physical properties that can be perceived as cosmetically appealing during the application phase.

Humectants, in combination with occlusive emollients, proved to be very important for sufficient moisturizing effects. Furthermore, the increased skin hydration results after application of Canoderm® highlights the difference in hydration efficiency between urea and glycerol (present in SC.c). The perception study did not emphasize any effect in sensorial properties that could be related to the moisturizing effect. However, small differences regarding tactile friction between Canoderm® and SC, could perhaps be related to increased skin hydration for skin samples treated with Canoderm®. This observation is in agreement with similar observations made by Lodén (Lodén et al., 1992) and Skedung (Skedung et al., 2016), however in neither case was skin hydration measured.

The choice of the right emollient composition is important for the sensorial properties of a cream. The results showed that the different emollient composition may be the explanation for the difference seen between the surfactant-based creams. Canoderm® and SC.c were ranked highest for softness. However, due to the variety of emollients used in the emollient mixture it was not possible to conclude which type of emollient that had a greater influence on the physical and sensorial properties. The amount of emollient used in the prototype creams (PC.c, SC, SC.c) was almost similar (28–30 wt%) while Canoderm® can be assumed to have lower emollient concentration with different surfactant composition (patent SE511551.C2).

The use of starch particles replacing traditional surfactants as emulsion stabilizers in a cream resulted in large differences in the perceived sensory properties, as well in the physical properties of the cream. These differences resulted in lower rank preference for sensory attributes important during the application phase of creams. Nonetheless, the overall sensorial benefit of the addition and use of starch particles as emulsion stabilizers could be related to the afterfeel sensation of creams. Generally unpleasant afterfeel attributes such as perception of a residual coating, stickiness and greasiness can be reduced and replaced by the sensation of a dry, powdery afterfeel. Similar results have been reported for inorganic particles in the residual oil film (Terescenzo et al., 2020), where the particles affected the properties of the residual film.

Interestingly, PC.c was not perceived as a thick cream during the application, although the rheological results showed higher viscosity and yield stress values than for SC.c. Furthermore, PC.c was not considered soft and smooth, and one explanation could be that the assessor's connected soft and smooth to the thickness and body of the cream, rather than the soft afterfeel sensation as a result of presence of particles. Instead, Canoderm® and SC.c were considered thick and soft, and this could be connected to presence of fatty alcohols among the surfactants, contributing to the body and thickness of the creams that can be perceived as soft, cushioning, and smooth.

Despite the low number of formulations tested in this study, the results clearly demonstrate the impact of replacing surfactants with stabilizing particles on the sensory and physical properties of a cream. We can conclude that the right combination of a thickener, humectant, emollient composition, and emulsion-stabilizer composition is needed in order to create a cream that can fulfill the stability and packing demands, and at the same time fulfill the sensory and moisturizing requirements. The use of starch particles to create a Pickering cream shows

great possibilities to design surfactant-free creams that can meet the demands for sensitive skin and sustainable skin care products with unique sensorial properties.

Our results further indicate that to fulfill the requirements of the participants in the sensory panel in this particular study, a formulator would need to design a cream that is soft upon application, and non-greasy in afterfeel, and with a good moisturizing effect. With the knowledge in hand, a combination of the moisturizing property and softness of Canoderm® with the non-greasy, non-sticky, powdery afterfeel of PC.c would be needed to fulfill consumer demands.

## 5. Conclusions

The results show that attributes related to the application phase such as thickness and spreadability can be predicted by means of rheological measurements, while tactile friction measurements on excised skin can be used to predict certain sensorial attributes related to the afterfeel sensation.

The type of emollients used, surfactant composition, as well as the presence of thickeners had the greatest impact on the difference in rheological and sensory properties among surfactant-based creams. Differences between a starch-based Pickering cream and surfactant-based creams were more evident in relation to the afterfeel attributes, and easily distinguished using tactile friction measurements.

A starch-based, surfactant-free Pickering cream with higher friction coefficient values ranked low for greasiness, stickiness, slipperiness, and softness.

### CRediT authorship contribution statement

**A Ali:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing – original draft, Writing – review & editing, Visualization, Project administration. **L Skedung:** Conceptualization, Methodology, Writing – review & editing, Formal analysis. **S Burleigh:** Formal analysis, Writing – review & editing. **E Lavant:** Investigation, Writing – review & editing. **L Ringstad:** Conceptualization, Methodology, Writing – review & editing. **CD Anderson:** Conceptualization, Methodology, Writing – review & editing. **M Wahlgren:** Conceptualization, Methodology, Supervision, Writing – review & editing. **J Engblom:** Conceptualization, Methodology, Supervision, Project administration, Writing – review & editing, Funding acquisition.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Acknowledgements

We are grateful to the Knowledge foundation (Sweden) for funding the project, and Johan Engblom also thank the Gustaf Th Ohlsson foundation (Sweden) for financial support. Speximo AB is acknowledged for providing starch particles.

### Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ijpharm.2021.121370>.

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