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“Innovative Characterization of Whipped Body Butters through Instrumental Analysis and Sensory Evaluation”

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1. Introduction

Whipped cosmetic products paid significant attention in the beauty industry due to their unique texture and sensory appeal. They are aerated cosmetic emulsions obtained by expanding a gas within the emulsion made of an oily phase dispersed in an aqueous phase. In the cosmetic field, whipped products are very sought after because they provide a very light texture that improves the sensoriality. Among cosmetic care products are body butters. They combine rich, nourishing ingredients like shea butter and oils, with gas, resulting in a product offering a light, airy consistency that contrasts with traditional body cares. The aerated emulsions are very ludic and offer an original sensory touch compared to the traditional products rich in fat and complicated to spread [1]. Due to its very airy texture, whipped products are very sensitive to external conditions such as transport and storage as well as temperature variations. The main challenge of cosmetics companies is to develop airy textures with a high-performance product that provides sensory pleasure during use and prolonged stability. Previous research intensely focused on whipped emulsion in the food industry and the roles of compounds to overcome instability issue and bring specific features to final products [2-4]. In the cosmetic field, there is a lack of knowledge about the development and physical and sensory characterization of whipped creams and the link between the components and products properties. Besides the fact that the whipping process must be carefully handled to achieve an optimal texture, an additional challenge lies in the characterization of the whipped product that is brittle by its nature. The analysis of this particularly fine and delicate airy texture requires to adapt the analysis methods.

This work explores the properties of a whipped product using conventional analysis techniques and adapting them to better characterize their specific features. The primary objective was to develop specific protocols for this unique aerated texture by adapting our traditional emulsion characterization techniques. The particularity of the aerated texture is that it is very difficult to handle without risking breaking its sensitive structure filled with gas bubbles. The analysis process was especially modified and optimized to facilitate the characterization steps without disturbing the product delicate structure.

The second objective is to understand the specific properties of whipped products using the optimized methods. Three studies were conducted to highlight the specific properties of whipped products and increase the knowledge on their behavior. The first study aims to demonstrate the impact of the whipping process, and how the incorporation of a gas modifies the physicochemical and sensory properties of the emulsion. The second objective of this study focuses on the kinetic follow up to better understand the long-term stability and microstructure evolution of whipped emulsions. The third part investigates the impact of raw materials (butter source and gelifying polymer concentration) regarding the firmness of the whipped product to better understand their role in the formulation and optimize the ingredients selection.

2. Materials and Methods

Whipped product

The studied product is a body butter with nourishing properties. It is obtained by whipping an initial oil-in-water emulsion -containing some fats (oils and butter) in the oil phase- with gas (nitrogen or air). The product density decreases from around 0.9 to around 0.6 after gas introduction.

Macrostructural evaluation

The observation of the samples was carried out in two ways: a first developed method allows to preserve the structure of the bubbles of the whipped product. The second method is more conventional and consists of the observation of the emulsions.

For the gas bubbles characterization, slices of whipped butter samples were observed under a binocular microscope to show the distribution of air bubbles in the heart of the sample. The slices of whipped butter were collected using a coring process. Coring process was developed on a texture analyzer using a butter cutting wire. The protocol consists of removing a piece of sample with a punch, then, from this piece of sample, a thin slice (2cm) was obtained by delicately cutting the piece of sample with the butter cutting wire.

For the characterization of the emulsions, the samples were examined at room temperature using Leica DM6 microscope. For samples preparation, the no whipped and whipped body butters were smeared on the microscope glass slide and covered by the cover slip. Images were taken with brightfield and polarized light with a x40 objective.

Microstructural evaluation

Rheology was used to evaluate the microstructure, through the viscoelastic properties. Amplitude sweep was conducted using an MCR302 rheometer (Anton Paar) equipped with a plate geometry (PP35/S) and a 2mm gap. Amplitude strain sweeps were performed from 0.001 to 1000% at a frequency of 1.0Hz and at 25°C. The storage (G′) and loss (G″) moduli in the linear viscoelastic region (LVR) were obtained.

Firmness of the whipped emulsions

A cone-penetration test was conducted to evaluate the firmness of whipped products using a texture analyzer TA.XTplus (Stable Micro Systems) with a 5kg force sensor equipped with P/30C probe. Penetration test (return to start) was performed at 1mm/s tests speed, with a penetration distance of 20mm. The trigger value for the start of the measurement was set to

0.50g. For the kinetic study, the whipped body butter was evaluated at 4 and 10 months from the whipping process. Tests were carried out in triplicate.

Calorimetry analysis

DSC thermograms were obtained with a differential scanning calorimeter DSC25 (TA Instruments). The samples were at first equilibrated at 20°C, then the heat-up step began from 20°C to 90°C at 2°C/min.

Sensory analysis

A sensory evaluation was conducted to objectively describe the texture. 16 trained experts characterized the whipped butters through 18 sensorial attributes following the descriptive methodology (profile). A first evaluation was conducted on the same batch of whipped butter after 4 and 10 months of ageing to study its evolution in time. The second sensorial evaluation was realized on the same formula before and after the whipping process (with and without gas addition) to better understand the sensorial impact of whipping. The statistical analysis was conducted using ANOVA and Student tests (alpha = 5%) and the results were interpreted using the p-value.

Design of Experiment DoE

A screening DoE (factorial matrix of 9 experiments: 2 parameters with three levels) was used to investigate the role of 2 ingredients on the whipped body butter firmness: the type of butter raw material (B1, B2, and B3) and the concentration of the gelifying polymer A (from 0.1% to 0.8%).

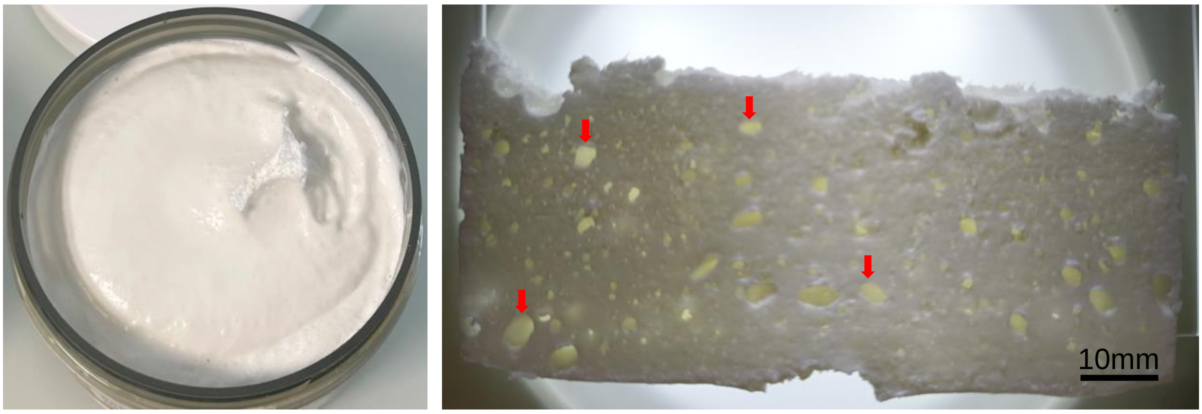
3. Results

1/ Characterization of whipped products: development and adjustment of protocols

The whipped products have a particularly potentially brittle structure and might be sensitive to transport and handling. A particular attention must be devoted to the characterization and sampling stages. Physical characterization methods need to be adjusted in a way to fit with this particular and new galenic formulation, with the aim of preserving the aerated emulsion structure. This chapter deals with the development of analytical protocols classically used on standard emulsions and then adapted to whipped products.

Macrostructural evaluation

The main characteristic of a whipped product is the presence of a gaseous phase in the form of bubbles in the emulsion. Figure 1(a) shows the picture of a whipped product in a 250mL jar. The whipped product has a smooth surface appearance, and bubbles are hardly visible. This representation is not sufficient to characterize the bubbles (size, number) of the whipped product located deeper in the emulsion. A coring protocol was developed to visualize deeper inside the whipped product. A 7x4x2cm^3 slice of whipped product was obtained and gently deposited on a glass slide as presented on Figure 1(b). The whipped product presents a three-dimensional network, with the presence of gas bubbles that has been incorporated, surrounded by the emulsion. Gas bubbles have a round and ovoid shape, and their size is between 1 and 5 mm (red arrows).



(a) (b)

Figure 1. Pictures of: (a) a whipped product in its container; (b) a slice of a whipped product.

Microstructural evaluation

The rheological analysis of the whipped product was carried out by adapting the analysis protocol to this type of emulsion. Conventionally, to determine the visco-elastic behavior of emulsions, deformation or frequency sweep is performed using plate/plate geometry with a gap between 0.5 and 1mm. Such protocol cannot be reliable with whipped products because a too small gap crushes the bubbles and considerably deforms the structure of the emulsion. To preserve the structure of the whipped products, the protocol was slightly adapted. The first adjusted parameter was the height of the gap. It was increased to 2mm to maximize the preservation of the product structure. The second adjusted parameter was the geometry surface (rough or sandblasted). This parameter must be taken into account to limit the effects of ejection/slippage. A rough geometry was used instead of the smooth sandblasted to limit the sliding phenomena. By adapting the classic rheological emulsion analysis protocol, the viscoelastic properties of whipped products were successfully evaluated.

Firmness of the whipped emulsion

Firmness analysis was carried out using an existing protocol for foam characterization with few modifications. Penetration force is the maximum force measured when the probe penetrates into the product, and it corresponds to the product firmness. Due to their very fragile texture, the firmness test on the whipped samples was carried out directly in their original jar (250mL, 90mm diameter). To avoid additional damage, the cylindrical (or hemispherical) probe mostly used for emulsions and gels, was replaced by a conical probe. The cylinder probe crushes the whipped product on the surface and deforms the bubbles, whereas the conical probe facilitates the penetration of the probe into the compacted and firm product. Penetration speed and distance have been also adjusted. A slow speed of 1mm/s enables the conical mobile to penetrate the whipped samples without breaking the nearby bubbles. An optimum penetration distance of 20mm was preferentially chosen to measure penetration forces at the center of the pack. By adapting the firmness analysis protocol, the texture of whipped samples was optimally characterized.

Calorimetry analysis

DSC analysis of whipped product is particularly delicate because the product sampling stage is unavoidable. With a conventional, non-whipped emulsion, the product is spread evenly on the bottom of the pan. For whipped product, the sample is simply dropped at the bottom of the pan to preserve the product's aerated structure.

Sensory analysis protocol adaptation

Traditionally, samples for sensory evaluation are repacked in neutral blinded jars to avoid any bias related to the packaging, the brand or any other visual cue aside from the texture. However, due to the sensitivity of the whipped butter, we couldn't repack the samples without risking an alteration of the sensorial properties. Therefore, the trained experts carried out the sensory evaluation directly in the finished product packaging to preserve the texture. Each volunteer was given one finished product for evaluation.

2/ Evaluation of the whipped product properties: physical and sensory characterization

This part aims to demonstrate the specific features of the whipped product. Its characterization using the previous adapted methods contributed to improve the knowledge and the understanding on its properties in terms of process, kinetic evolution, and composition.

Impact of the whipping process

The objective of this part is to highlight the influence of the gas incorporation process on the whipped product properties. The physical and sensory properties of the body butter emulsion were compared before and after the incorporation of the gas, to show the benefits of the process on the texture.

Firmness protocol was performed on the emulsion with and without gas incorporation. Figure 2 showed that the firmness of the emulsion (the maximum force measured during penetration) before whipping (green line) was about 120g. After the whipping process (blue line), it decreased until around 79g. The incorporation of gas into the emulsion facilitates the probe penetration and induces a reduction of the firmness for the whipped product. The reduction of the firmness can be useful to change the touchy perception of an emulsion and for making it lighter.

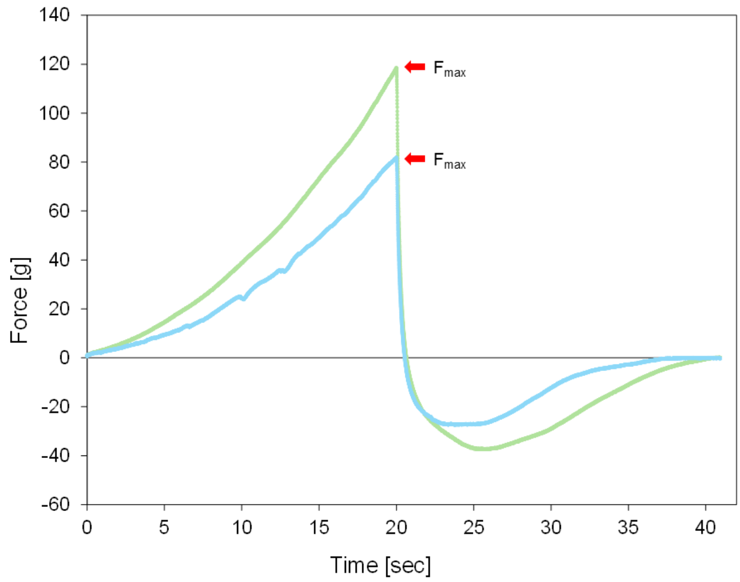


Figure 2. Evaluation of the maximum force of whipped (blue line) and not whipped (green line) body butters.

The sensory evaluation of the two samples showed that both profiles were very close as presented in Figure 3.

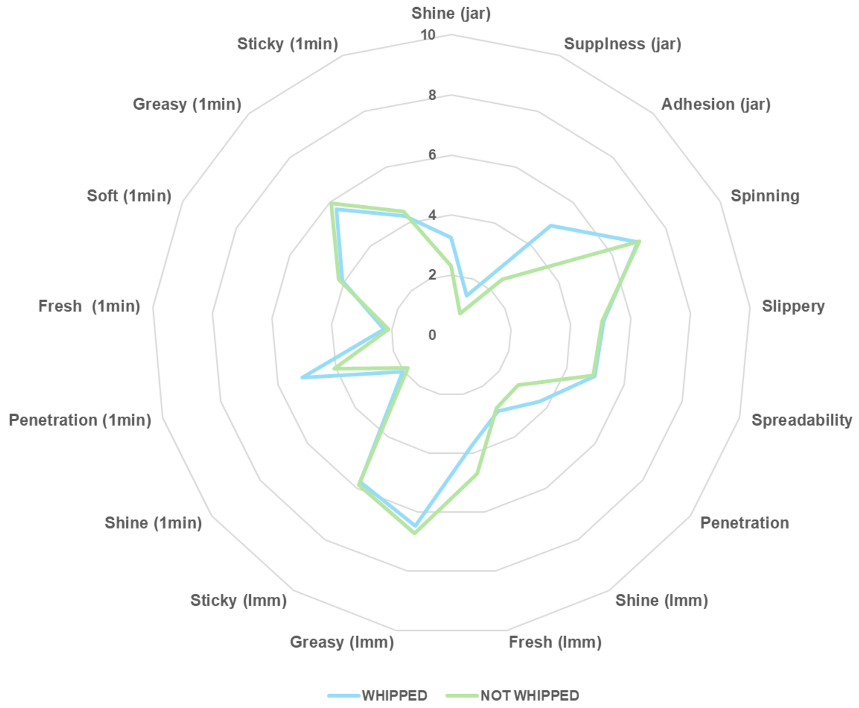


Figure 3. Sensory profiles comparison between whipped (blue line) and not whipped (green line) body butters.

The statistical analysis showed no significant difference for all the attributes evaluated during the application, immediately after application and 1 minute after application. Nevertheless, the statistical comparison displayed a significant difference on the three attributes evaluated in the jar and during the pick-up: brightness (jar), suppleness (jar) and adhesion (jar). The whipped texture is significantly brighter, more supple and with a higher adhesion than the texture without gas incorporation.

The incorporation of a gas phase in the emulsion seems to impact the visual and pick-up of the whipped product by increasing its brightness, suppleness and adhesion for a new consumer experience. The attributes evaluated after application showed no significant difference and this may be related to the transformation of the texture when applied and the gas being expelled from the texture.

The rheological behavior was evaluated on the emulsion before and after the whipping process. Tan δ (G''/G') values were lower than 1, meaning that the elastic behavior is more pronounced than the viscous behavior. No significant differences in tan δ values were observed between the two samples. The dynamic moduli G' (storage modulus) and G'' (loss modulus) are plotted as functions of shear deformation. The limit of the viscoelastic domain is the same and equal to 0.10% suggesting that the product is easily deformable. However, at the intersection of the modules (G'=G''), the value of the modulus is higher for the whipped emulsion suggesting a greater level of structuration than the no whipped emulsion. Additionally, G′ and G'' values of the whipping emulsion are superior to the no whipped emulsion. The whipping process induced a slight increase of the compacity due to the incorporation of the gas that modifies the microstructure of the emulsion.

Calorimetry analysis was performed to check if the whipping process could modify the structure of the emulsion through the crystallization of the fats. Figure 4 shows endothermic peaks corresponding to the melting of the raw materials from the oily phase such as butter, fats, and oils. The thermograms showed that there was no significant difference between the emulsions before and after the whipping process. The incorporation of the gas into the emulsion does not involve crystallization of the fats in the bulk. Also, identical thermograms suggested that there is no degradation of the product after the whipped process.

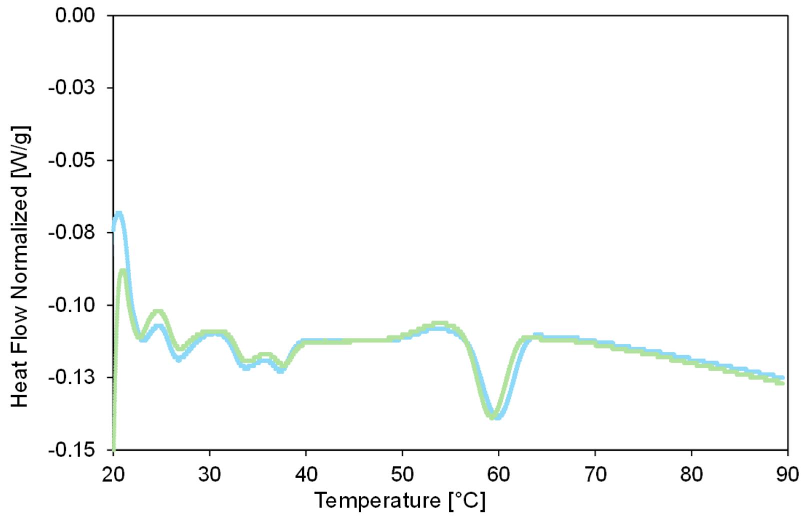


Figure 4. Thermograms of whipped (blue line) and not whipped (green line) body butters.

Figure 5 shows the micrographs of the body butter before the whipping process and the whipped body butter. The two samples showed that the emulsion is a typical oil-in-water emulsion with oil droplets dispersed in the aqueous phase. The droplet size of the emulsion before and after the whipping are the same suggesting that the whipping process does not modify the droplets size of the emulsion.

Polarized light microscopy was used to reveal the microstructure of the emulsion. The polarized light micrographs on both no whipped and whipped body butters manifested a light reflection (birefringence), indicating the presence of a structured crystalline gel network that might prevent the fat globules coalescence. After the whipping process, the birefringence was also detected. This observation supported the fact that the whipping process does not induce the network destructuration after incorporation of the gas phase.

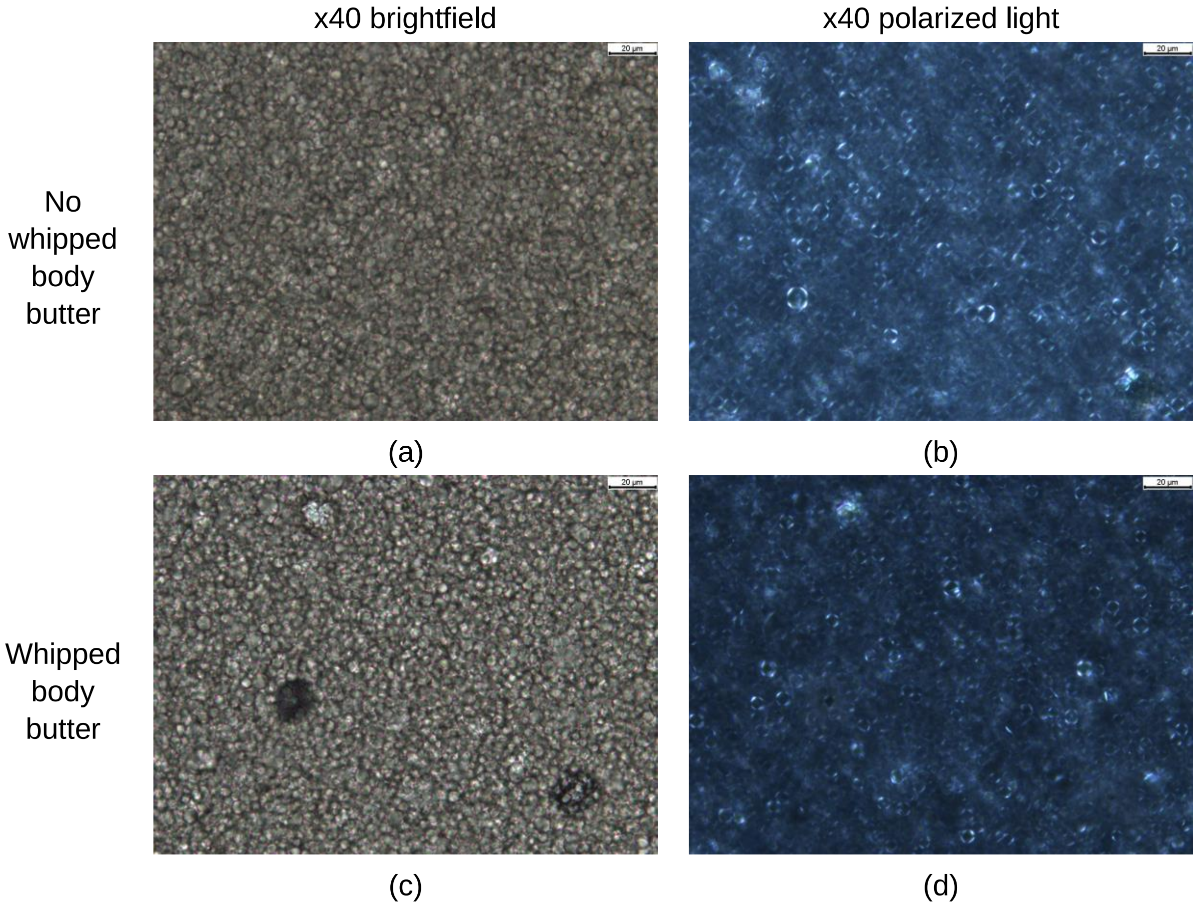


Figure 5. Micrographs (a-b) of the body butter before the whipping process and micrographs (c-d) of the whipped body butter.

Kinetics studies of whipped emulsions

Kinetic studies were carried out on two batches of the whipped body butter to characterize the firmness evolution against time. The firmness was evaluated on the two batches between 2 and 10 months and the results are presented on Figure 6. At 3 months the firmness was around 49g, and at 10 months, it was around 90g for the first batch (black dots). For the second batch (red squares), at 2 months, the firmness was around 47g, and at 10 months, it was around 107g. The firmness evolution of the two batches was quite similar. It increased along the time until the 8 months where it slowed to reach a plateau.

Furthermore, we conducted a sensory evaluation at 4 and 10 months on one of the whipped body butters that had the highest gap of firmness between two temporalities, to evaluate the impact of time on this texture. The results showed no significant difference on all the attributes between a "young" (4 months after fabrication) and a more “mature” sample (10 months after fabrication).

The evolution of the firmness measured by the texture analyzer has not been detected by the trained panelists. The expert panel does not have the same sensitivity compared to the instrumental technique and here it does not perceive a difference between the young and the mature whipped body butter.

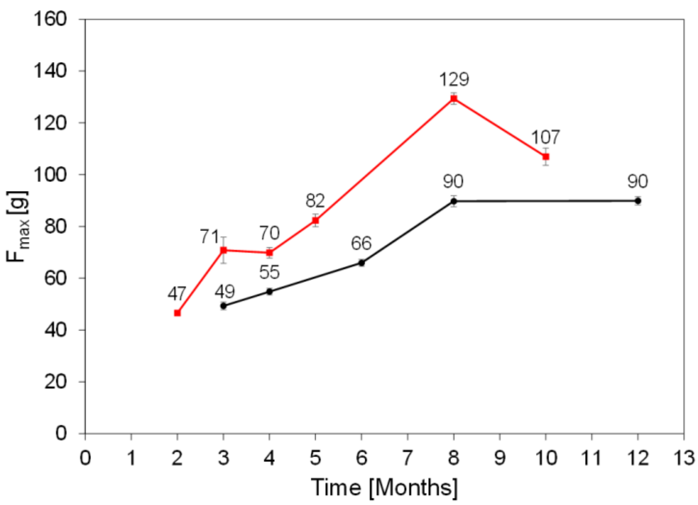


Figure 6. Firmness evolution of two batches (black dots and red squares) of whipped body butters.

Impact of raw materials

The impact of two ingredients categories: the type of butter and the gelifying agent concentration regarding the firmness of the whipped product have been investigated through a design of experiment. Figure 7 shows that the firmness of the whipped product decreases as the concentration of the gelifying agent increases, independently of the selected butter. This effect was particularly pronounced with the butters B1 and B2. At 0.1% of gel concentration, the butter type mainly influences the level of firmness, especially between B3 and the 2 other butters B1 and B2. At 0.8% of gel concentration, the firmness is less significant between the 3 butters and the gelifying agent brings more suppleness to the product.

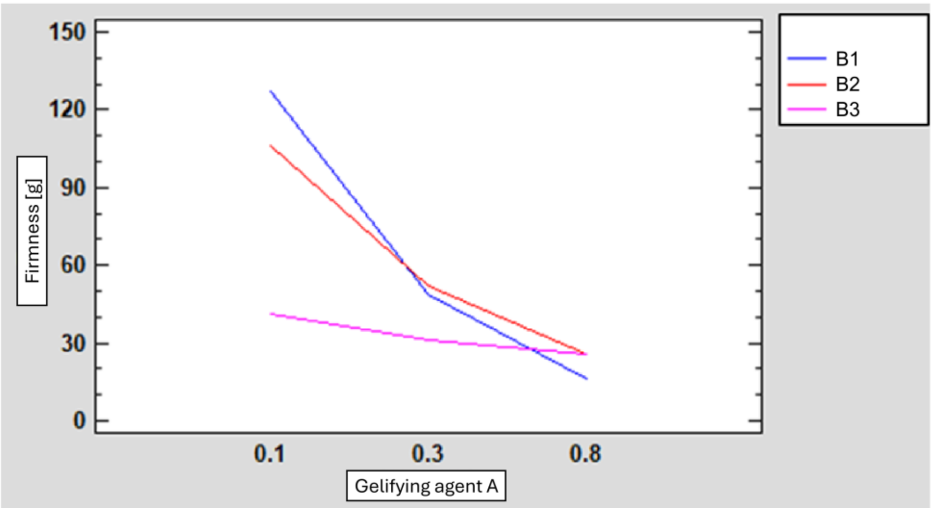


Figure 7. Evolution of firmness against gelifying agent concentration and butter type.

4. Discussion

Whipped products are innovative products with specific texture and sensorial properties. However, the characterization methodology of these products is different from the conventional oil in water emulsions. The analytical protocols were adapted to this particular gas containing galenic. An innovative macrostructural characterization has been developed for the visual observation of the gas bubbles on a slice obtained by a coring method. The main advantage of this technique is that it is possible to assess the gas incorporation into the emulsion without crushing the whipped structure. To improve this protocol, quantification of bubble sizes by image analysis could be considered. This coring protocol could also be useful subsequently if we want to tailor the volume of the whipped product to take it out from its container and perform some analysis without affecting irreversibly the sample.

The firmness evaluation was performed directly in the final container in order to free sampling or reconditioning steps and avoid bias in the measurement. The adaptation of the probe was also taken into account for the optimization of the method. For whipped products, the conical probe was preferred instead of hemispherical probe (conventionally used to imitate a finger pressing) because the results can be repeated with a good accuracy without crushing the structure during penetration. The determination of the linear viscoelastic domain in rheology was optimized by increasing the gap height and using a rough geometry that avoids sliding effects. Sampling step is important to delicately put the whipped emulsions between the parallel plate for rheology and deposit whipped emulsions in the pan for calorimetry analysis.

The comparison of the emulsion before and after the whipping process showed that the firmness level of the whipped emulsion is lower, which is consistent with its sensory analysis, where it was perceived as more supple compared to the no-whipped emulsion. At the same time, the rheology results demonstrated a slightly increase of the stiffness in the whipped emulsion due to an increase in G' and G'' values, and thus a slightly higher product compacity. Micrograph analysis and thermograph analysis supported the fact that the emulsion before and after whipping process have equivalent droplets size, and a similar structure. Although the whipped formula is more supple and less firm, its microstructure was slightly impacted keeping the integrity of its structure and the size of the emulsion droplets.

The kinetic study highlighted the behavior of both whipped samples to become increasingly firm over several months. The firmness gap was analyzed in a sensory study, but no significant difference was found between a young sample and a mature sample. It is interesting to note that a firmness gap of approximately 30g was not perceived by the panel for all the descriptors analyzed.

The type of butter and the gelifying agent to be incorporated into the fat phase during formulation are key raw materials to tune the final firmness of the whipped product. The gelifying agent can reduce the firmness of the butter more or less efficiently depending on its concentration and also on the type of butter used in the initial emulsion. The role of these two raw materials has been highlighted to understand their impact on the textural properties of the final whipped product.

5. Conclusion

This study provided an innovative adaptation of conventional analysis techniques for a particularly sensorial category of whipped products. Physical and sensory methods were fitted to this new galenic by preserving the aerated emulsion structure and to demonstrate their specific features and microstructure evolution against time. The study raises the knowledge and the understanding on whipped products properties and especially how ingredients combination and production process can be tailored to enhance consumer sensory experiences.

[1] K.F. Dabo, C. Chèné, A.-L. Fameau, R. Karoui, Molecules, 29, 5933, 2024.
[2] R. K. Thakur, C. Vial, G. Djelveh, Chemical Engineering and Processing: Process Intensification, 47, 3, 474-483, 2008.
[3] K. Ihara, M. Hirota, T. Akitsu, K. Urakawa, T. Abe, M. Sumi, T. Okawa, T. Fujii, Journal of Dairy Science, 98, 5, 2875-2883, 2015.
[4] M. Grossi, M. Zhao, J. Rao, B. Chen, Trends in Food Science & Technology, 153, 104752, 2024.