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“Potentializing and accelerating hair care formulations through design of experiments powered by sensory feed-back”

Gabriel Rocha[1], Liliane Duarte[1], Deborah Barros[1], Bruna Conrado[1], Gwendoline Shipalana Mbatha[1], Nathalie Lorencini[1], Natalia Shecaira[1], Pedro Aprigliano[1].

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1. Introduction

The cosmetics industry is constantly evolving, driven by the relentless pursuit of products that meet consumers' ever-increasing expectations for efficacy and sensory experience. Developing new hair care formulations presents significant challenges due to the complex interplay of factors influencing sensory perception, such as texture, shine, softness, and fragrance.[6]

Traditionally, Design of Experiments (DOE) has been a fundamental tool for optimizing formulations, using instrumental data to guide the process [1,2]. However, while instrumental analysis provides valuable information about the physicochemical properties of formulations, it often fails to capture the subjectivity and complexity of human sensory perception [3,4]. The growing demand for cosmetic products that deliver memorable sensory experiences underscores the need for methodologies that integrate sensory analysis as a core component of the development process [4].

This study proposes an innovative approach that places sensory analysis at the heart of DOE, using sensory data as the primary input for optimizing leave-in hair care formulations. Our main objective is to demonstrate that integrating sensory analysis into DOE accelerates the development process and, more importantly, results in formulations with superior sensory performance, aligned with consumer expectations[4,5].

This approach has the potential to go further in cosmetic product development, enabling the creation of formulations that provide unique and differentiated sensory experiences. We will explore the application of this methodology in the context of leave-in hair formulations, a segment where sensory perception plays a crucial role in consumer product acceptance [3].

2. Materials and Methods

This section details the materials and methods used in this study.

2.1 Formulation Preparation

The preparation of the formulations used in this test required samples of all raw materials under consideration for the project. Essential equipment included a Rayneri laboratory mixer, a heat plate, and a pH meter for measuring pH levels.

2.2 Hair Sample Selection and Preparation

Virgin Caucasian hair tresses were obtained from a specialized supplier. The tresses were washed with a standardized neutral shampoo (approximately 2.7g of shampoo per tress), then detangled with a fine-tooth comb until completely detangled. The tresses were subsequently air-dried before testing. This standardized procedure ensured the removal of any cosmetic product residues or impurities that could interfere with the sensory analysis results.

2.3. Experimental Design Space Definition and Raw Material Selection

Before generating the formulations using the DOE system, the experimental design space was defined, considering the raw materials and their respective concentration ranges. The selection was based on prior knowledge acquired in previous internal studies, considering their influence on the sensory attributes, such as softness, shine, detangling, and frizz control. The selected raw materials included surfactants, oils, emollients, and polymers. A carefully defined design space is crucial to ensure that the generated formulations are viable and meet the final product requirements.

2.4. Generation of 26 Formulations by the DOE Platform

After defining the design space, an internal software was used to generate the 26 leave-in formulations. This platform employs machine learning algorithms and statistical methods to propose formulations that efficiently explore the design space, maximizing the information gained from a limited number of experiments, aiming to identify formulations with specific sensory properties, and utilized a space-filling algorithm to ensure uniform distribution of the formulations within the design space.

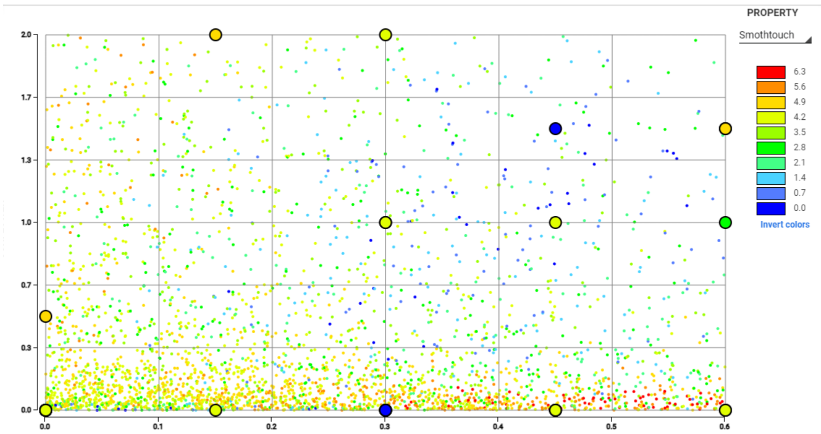


Figure 1. This picture illustrates the distribution of some of the generated formulations by the system, in a graph depicting the varying concentrations of two specific raw materials.

2.5 Sensory Analysis

A sensory analysis expert with extensive experience in hair product evaluation conducted the sensory tests for this study. The hair tress evaluation considered the attributes of transformation, softness, and coverage, which were measured during the application of the hair product. Tests were performed under controlled temperature (25°C ± 2°C) and relative humidity (50% ± 5%) conditions. For each leave-in formulation, the expert received a precise amount (0.41g ± 0.1g) for application to a virgin hair tress, following a standardized application. Sensory evaluation employed a 5-point scale, where 1 represented the lowest intensity and 5 the highest intensity for each attribute.

Grades	
High	5
Medium +	4
Medium	3
Medium -	2
Low	1

Application attributes:
Transformation
Smooth touch
Coverage

Figure 2. Sensory attribute rating scale

2.7 Design of Experiments (DOE) and Optimization

The collected sensory data were analyzed using DOE software. A statistical model was built to correlate the raw material concentrations with the evaluated sensory attributes. Based on this model, six optimized formulations were identified, aiming to maximize overall sensory performance. These formulations were then produced and subjected to a new sensory evaluation with the sensorial expert for final formulation selection.

2.7 Comparison with Benchmarks

After DOE optimization, the selected formulation was compared to an internal benchmark and a commercially available reference product. The in vitro sensory evaluation, using standardized hair tresses, followed the previously described protocol, enabling direct comparison of the new formulation's performance against established products.

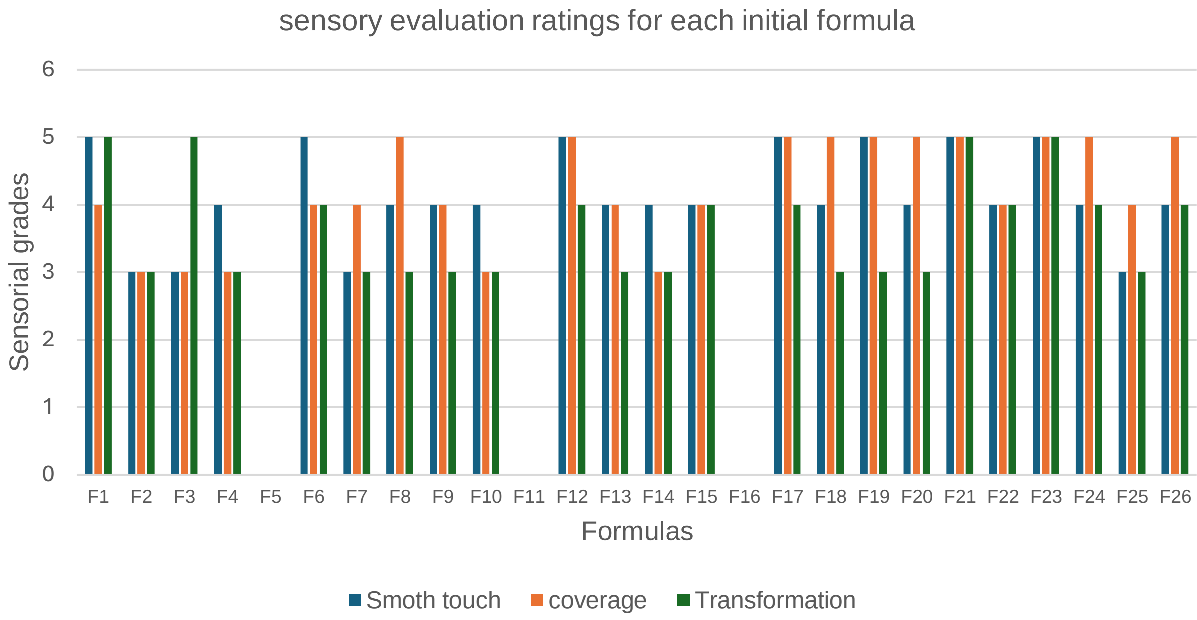
To validate these preliminary results and confirm the optimized formulation's performance under real-use conditions, a comparative in vivo test (N=8) was conducted. Eight volunteers had their hair divided into two sections, with the optimized formulation applied to one half and the benchmark reference product (either internal or commercial) applied to the other half. Application was performed by sensory analysis experts, following a standardized protocol ensuring the same product quantity and application technique on both halves of the head. This analysis is considerably more robust than the standard hair tress analysis. As a test conducted on volunteers' heads, it allows for the evaluation of various attributes involved in the usage journey of a hair styling cream, such as application attributes, immediate effects, and effects after hair drying. This comparative in vivo test aimed to verify whether the DOE-developed formulation achieved, in real-world use, performance standards comparable to established market products.

3. Results

This section presents the results obtained in this study, organized into subsections to facilitate understanding and data interpretation. The results of the sensory analysis, formulation optimization via DOE, and comparison of the final formulation with benchmarks will be described.

3.1 Initial Sensory Analysis of the 26 Formulations

The initial sensory analysis of the 26 leave-in formulations revealed a wide variation in the perception of the sensory attributes evaluated by the expert panel. Graph 1 presents the distribution of scores assigned to each formulation for transformation, softness, and coverage. Some formulations performed better in certain attributes, while others excelled in different ones, demonstrating the complex relationship between formulation composition and sensory perception. For example, formulations F1, F6, and F18 scored highest in softness, while F8, F12, and F17 excelled in coverage. Formulations F2, F21, and F23 showed the best results for transformation. Conversely, formulations 5, 11, and 16 did not form stable emulsions.

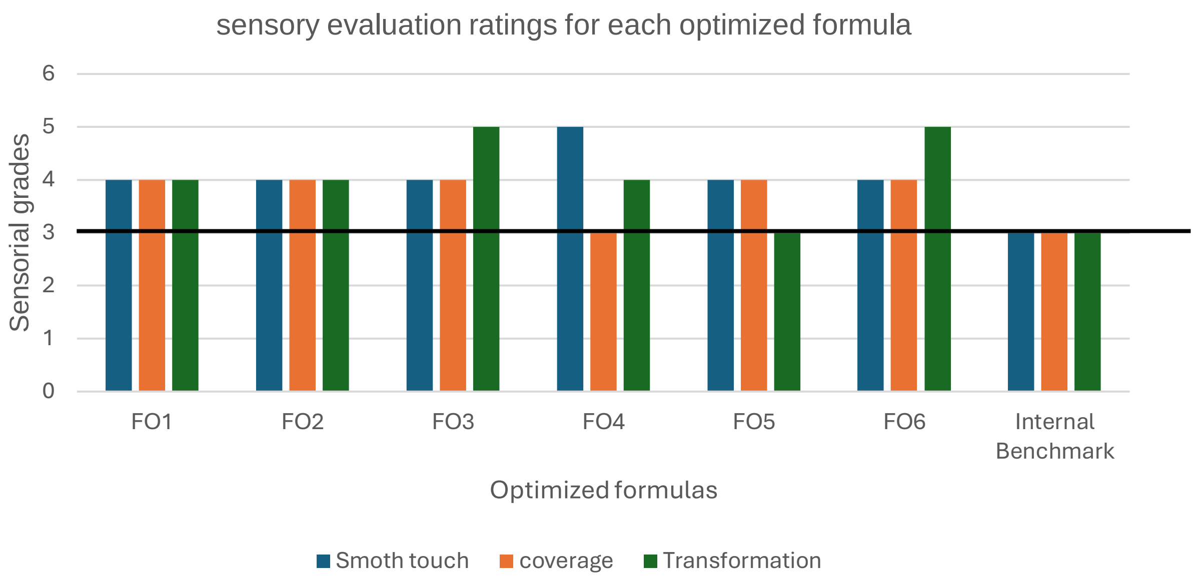


Graph 1. Sensory evaluation of the 26 initial formulations produced for DOE sample space population.

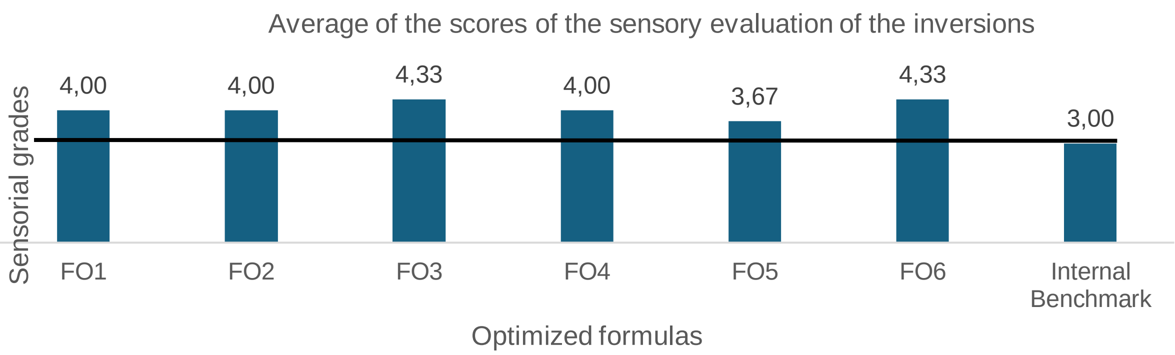
3.2 Formulation Optimization via DOE

Based on the initial sensory analysis data, the DOE model suggested six new optimized formulations (FO1 to FO6), aiming to maximize overall sensory performance. In this context, these formulations were then produced in the laboratory according to the previously described process.

Graph 2 compares the sensory performance of the optimized formulations against the internal benchmark. A clear increase in average scores for most sensory attributes is observed in the optimized formulations, indicating the effectiveness of the DOE model in guiding formulation optimization, as shown in graph 3. Formulations FO3 and FO6 exhibited the best overall sensory performance, considering the average of the evaluated attributes, these formulas perform on average 44% better than the benchmark. The formulation FO6 was discontinued due to its instability.



Graph 2. Sensory evaluation of the 6 optimized formulations suggested by the DOE.



Graph 3. The Average of the sensory evaluation, considering the 6 optimized formulations suggested by the DOE.

3.3 Comparison with Benchmarks

After testing the strands, we decided to proceed with FO3 for in vivo testing**.** The optimized formulation FO3 was compared with the chosen benchmark and a commercially available reference product in a comparative in vivo test (N=8). Tables 1 and 2 present the results of this comparison. FO3 demonstrated better performance compared to both benchmarks across various attributes throughout the style cream usage journey. For instance, attributes such as transformation, definition softness, shine, and frizz control showed better results than the reference products.

FORMULAS	TESTED FORMULA: *FO3* REFERENCE VS External Benchmark
CPP APP	> Transformation speed
WET HAIR	>> Coverage > Smooth touch
DRY HAIR BEFORE HARD EFFECT	> Definition < Frizz
DRY HAIR AFTER HARD EFFECT	> Definition > Shine > Smooth touch > Coverage > Softness << Frizz

Table 1. Pure comparative evaluation between FO3 and a market product, illustrating the sensory attributes with the best performance for the formula, using FO3 as a reference.

FORMULAS	TESTED FORMULA: *FO3* REFERENCE VS Internal Benchmark
CPP APP	> Transformation
WET HAIR	>> Definition > Smooth touch > Coverage > Softness
DRY HAIR (before hard effect)	> Definition < Frizz
DRY HAIR	>> Smooth touch > Definition > Shine > Coverage < Frizz

Table 2. Comparative evaluation between FO3 and an internal benchmark, illustrating the sensory attributes with superior performance for the optimized formulation, using FO3 as a reference.

4. Discussion

The results of this study demonstrate the potential of integrating sensory analysis with Design of Experiments (DOE) for the development of optimized hair care formulations. Formulation FO3, resulting from this methodology, exhibited superior sensory performance compared to both the internal and market reference benchmarks. This superiority is primarily reflected in attributes such as transformation, softness, shine, and frizz control, which are considered key purchase drivers in the leave-in hair care category. Our findings corroborate previous studies demonstrating the importance of sensory analysis in cosmetic product development. In the present study, optimizing ingredient concentrations via DOE, guided by sensory data, led to a significant increase in the perception of these attributes in formulation FO3.

The proposed methodology offers significant advantages over the traditional approach to cosmetic product development, which often relies on time-consuming and expensive instrumental and in vivo tests that often lack the agility demanded by today's market. By incorporating sensory analysis as a primary input in DOE, we significantly reduced development time, while also achieving a formulation with superior sensory performance. This more efficient and consumer-centric approach has the potential to accelerate innovation in the cosmetics industry, enabling the development of products that meet increasingly sophisticated consumer sensory expectations.

The funnel-shaped evaluation strategy, utilizing in vitro sensory analysis integrated with DOE, followed by only one comparative in vivo test. In similar projects within the category, prototype refinement typically requires up to 20 separate in vivo tests. In the model presented in this article, the ideal formulation was achieved with only one comparative in vivo test, significantly optimizing resource utilization and accelerating the project timeline. This drastic reduction in the number of tests demonstrates the efficiency of the proposed methodology, which prioritizes in vitro sensory analysis as a guide for DOE, enabling more precise and efficient selection of candidate formulations before in vivo testing.

However, it is important to acknowledge the limitations of this study. Sensory analysis, being subjective, can be influenced by factors such as individual evaluator experience and environmental conditions. To minimize these effects, we employed a highly trained sensory expert and conducted evaluations under controlled conditions. Future studies could explore the application of this methodology with different hair tress types and consumer panels to compare with the results obtained from expert analysis. Furthermore, it would be valuable to investigate these alternative methods with other product types, such as rinse-off hair care products like conditioners, masks, and shampoos, or even skincare or sun protection products, broadening the scope of optimization.

5. Conclusion

This study demonstrated the effectiveness of integrating sensory analysis with Design of Experiments for developing leave-in hair care formulations with better sensory performance. Formulation FO3, obtained through this methodology, outperformed benchmarks in key attributes like transformation, softness, shine, and frizz control. The proposed approach significantly reduced development time while ensuring a final product better aligned with consumer sensory expectations, as demonstrated in further consumer testing that followed this study. Internal product development can take up to 20 in vivo tests to refine the formula and surpass benchmark performance, requiring only one in vivo test, reducing workload, and accelerating project timelines.

Integrating sensory analysis with DOE represents a simple yet promising advancement for the cosmetics industry, with the potential to accelerate innovation and the development of more effective and enjoyable-to-use products. Future studies should explore the application of this methodology across other cosmetic product categories and with diverse consumer groups to validate and expand upon the results presented here.

6. References

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