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Encapsulation of pequi oil optimize by the spray drying process and application in a leave-in formulation for curly and straight hair

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1. Introduction

Hair damage is a common concern in cosmetic science, especially when caused by chemical processes such as bleaching, which can compromise the structural integrity of the hair fiber. This leads to increased porosity, reduced strength, dryness, and lack of shine, particularly in hair types already prone to fragility, such as curly hair [1]. As consumer demand shifts toward more natural and sustainable products, the use of plant-derived ingredients has become increasingly relevant in hair care formulations.

Among these natural actives, pequi oil (*Caryocar brasiliense* Cambess.; Sisgen: A15D96E), extracted from a fruit native to the Brazilian Cerrado, is notable for its high content of oleic and palmitic acids. These components offer emollient, antioxidant, and hydrating properties, making pequi oil a promising candidate for hair restoration and protection, particularly in damaged hair [2].

However, the direct use of vegetable oils in formulations presents challenges related to instability, such as oxidation and undesirable sensory characteristics. To overcome these limitations, liposomal encapsulation has proven effective in protecting sensitive compounds and enhancing their delivery to the hair fiber [2,3]. Additionally, converting liposomal dispersions into a powder form through spray drying enhances stability, handling, and incorporation of active ingredients into final cosmetic formulations [2,4].

To ensure the reproducibility and scalability of the spray drying process, the use of a Design of Experiments (DoE) approach allows for the systematic optimization of key process parameters. This strategy has been demonstrated to enhance product quality while concurrently facilitating industrial implementation through a data-driven decision-making process [5-7].

Evaluating the efficacy of cosmetic formulations on standardized hair tresses - matched for weight, length, and damage level - enable consistent comparison between treatments and reliable analysis using instrumental techniques such as tensile strength, combability, and texture [1,8].

Despite increasing interest in natural ingredients and encapsulation technologies in hair care, few studies have assessed their performance across different hair types. Evaluating

efficacy in both straight and curly hair is crucial, as these textures respond differently to damage and product performance, which directly impacts consumer satisfaction. In this context, the present study aimed to encapsulate pequi oil, optimize the spray drying process using experimental design, incorporate the dried liposomes into a leave-in formulation, and evaluate its efficacy on curly and straight hair tresses. Finally, this study combined advanced delivery systems with robust, quantitative efficacy testing, contributing valuable insights to the development of innovative, inclusive, and sustainable cosmetic formulations.

2. Materials and Methods

Liposome Characterization

The liposomes were characterized in terms of pH, particle size, polydispersity index (PdI), and zeta potential. Measurements were performed 24 hours after preparation. Particle size and PdI were assessed by Dynamic Light Scattering (DLS) using a Zetasizer Nano ZS90 (Malvern Panalytical, UK), with samples diluted 1:1000 (w/w) in Milli-Q® water. Zeta potential was analyzed by electrophoretic mobility under identical dilution conditions to evaluate colloidal stability [2].

Spray Drying and Experimental Design

Liposome dispersions were dried using a Lab-Plant SD-05 spray dryer. The process was optimized using a 3^2 full factorial Design of Experiments (DoE) with three central points. The independent variables were inlet temperature (80, 110, and 140 °C), carrier-to-liposome ratio (R = 0.28, 1.08, and 1.87), and maltodextrin content (0%, 50%, and 100%). It is important to note that the absence of maltodextrin (0%) corresponds to the use of Arabic gum as the drying adjuvant.

Drying parameters included a feed rate of 4 g/min, atomization pressure of 1.5 kgf/cm², and air flow of 60 m³/h with atomizing air at 17 L/min. The dried powders were collected and stored in hermetically sealed containers for further analysis [4].

The following properties were evaluated: drying yield, moisture content, water activity, Carr index, and Hausner ratio, which are critical indicators of powder stability and flowability. ANOVA was used to assess the significance of process variables, considering a 95% confidence level.

Development of the leave-in formulation

The leave-in formulation was developed using a base composed of conditioning agents, humectants, preservatives, and water, and were added or not (vehicle) with 1% of free pequi oil, 8% of empty dried liposomes (LB), or 8% of dried liposomes containing pequi oil (LPO). The concentration of pequi oil and liposomal powders was analyzed to ensure compatibility with the formulation components and maintain desirable sensory and functional properties.

To ensure formulation integrity, preliminary stability tests were performed over a 90-day period. These included assessments of pH, appearance, odor, and homogeneity, enabling the identification of any signs of phase separation, color change, or olfactory instability under controlled storage conditions. All formulations remained stable throughout the test period.

Hair Tress Evaluation Protocol

Standardized curly and straight human hair tresses (3 g, 15 cm in length) were used for all experiments. The tresses were first washed with a 3% sodium laureth sulfate solution, rinsed with water, and dried using a hairdryer at a warm temperature. Afterward, the tresses were stored in a controlled environment (25 ± 2 °C, 50 ± 5% relative humidity) for 24 hours.

The bleaching process involved preparing a mixture of bleaching powder and 30-volume hydrogen peroxide in a 1:2 ratio (45 g of powder to 90 g of H_2O_2). The mixture was applied uniformly to the entire length of each tress, which was then wrapped in aluminum foil and left to process for 45 minutes. Following this, the tresses were rinsed under running water for one minute, washed with neutral shampoo and dried using warm air.

Subsequently, the leave-in formulations were applied at a standardized amount of 0.075 g of product per gram of hair, according to each experimental group. After treatment, the tresses were evaluated using instrumental analysis to assess softness and tensile strength, enabling a quantitative comparison of the effects of the formulations on bleached hair.

The tresses were categorized as described in Table 1.

Table 1. Hair tresses and the following treatment.

Code	Hair Type	Treatment Stage	Description
CH1	Curly	-	Control – non-bleached
CH2	Curly	-	Control – bleached
CH3 AT	Curly	After Treatment	Treated with vehicle
CH4 AT	Curly	After Treatment	Treated with vehicle + free pequi oil
CH5 AT	Curly	After Treatment	Treated with vehicle + empty liposomes
CH6 AT	Curly	After Treatment	Treated with vehicle + liposomes containing pequi oil
SH1	Straight	-	Control – non-bleached
SH2	Straight	-	Control – bleached
SH3 AT	Straight	After Treatment	Treated with vehicle
SH4 AT	Straight	After Treatment	Treated with vehicle + 1% free pequi oil
SH5 AT	Straight	After Treatment	Treated with vehicle + 8% empty liposomes (LB)
SH6 AT	Straight	After Treatment	Treated with vehicle + 8% liposomes containing pequi oil (LPO)

Instrumental measurements

Tensile strain

To evaluate the mechanical properties of the hair, 20 hair fibers were obtained from the hair sample. The tensile strength test was performed using a Texture Analyzer TA.XT Plus® (Stable Micro Systems, Godalming, UK), equipped with Exponent 3.0.5.0 software. The hair fibers were fixed at a span of 250 mm and subjected to tension at a constant speed of 300 mm/min, using a load cell of 10 N, until the point of rupture. The resulting stress–strain curves were recorded to determine the tensile strength values of the samples [8].

Softness

The softness of the hair tresses was also evaluated using the Texture Analyzer TA.XT Plus® (Stable Micro Systems, Godalming, UK). This test involved applying a controlled deformation force to the tress to measure its flexibility and tactile quality. The deformation parameters were standardized, and the results were used as quantitative indicators of hair softness after treatment .

Statistical analysis

The DoE experiments were analyzed using the software Statistica 14.0.1. After data collection, analysis of variance (ANOVA) was performed to determine the significance of the effects of the studied factors on the response variables. Replicates at the central point were used to estimate experimental error. A p-value < 0.05 was considered statistically significant. The Shapiro–Wilk test was used to assess the normality of the data distribution. To compare the initial characteristics of the samples, unpaired statistical tests were applied. Depending on data distribution, either the Student's t-test (for normally distributed data) or the Mann–Whitney test (for non-normal distributions) was used. For the hair tests, the statistical analysis was

performed using the software Graph Pad Prism 9.0. The Shapiro-Wilk test was used to assess the normality of the samples. For normal distribution, one-way ANOVA with Tukey's post-test was applied, and for non-normal distribution, the Kruskal-Wallis test was applied with Dunn's post-test. A p-value ≤ 0.05 was adopted as the threshold for statistical significance.

3. Results

The liposomal formulations exhibited an average particle sizes of 280.08 nm for empty liposomes (LB) and 327.87 nm for liposomes containing pequi oil (LOP), with a statistically significant difference between them ($p < 0.0001$). The PdI was also significantly different, with values of 0.20 for LB and 0.32 for LOP ($p < 0.0001$), both indicating good size homogeneity. Zeta potential measurements showed good eletrical stability, with average values of –28.43 ± 0.51 mV for LB and –29.87 ± 0.97 mV for LOP, remaining near the ±30 mV.

Spray drying yield ranged from 54.33% to 73.29%, with higher yields associated with increased drying temperatures (140 °C) and the use of pure maltodextrin at lower R ratios. Surface response plots (Figure 1) showed that gum arabic performed better at 80 °C, while maltodextrin was more effective at 110 °C and 140 °C.

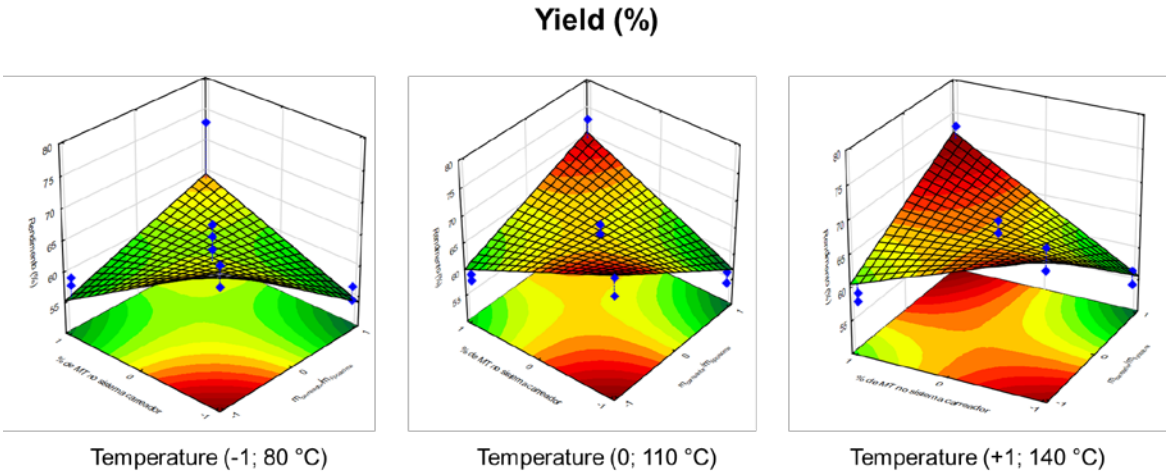

Figure 1. Response surface graphs showing the effect of the % of matrodextrin in the carrier system and the ratio between the mass of the carrier and the mass of the liposome (R) on the yield (%) of the drying process at different temperatures.

The flowability and compressibility values obtained from the Hausner ratio (HR) and Carr index (CI) were also dependent on formulation and temperature. Values below 1.25 for HR and 0.18 for CI were associated with powders obtained under higher drying temperatures and pure maltodextrin. At 140 °C, the lowest FH and IC values were observed for the formulation with pure maltodextrin and a low R ratio, indicating excellent powder properties.

Moisture content and water activity (a_w) showed no statistically significant differences among conditions ($p > 0.05$), with a_w values ranging from 0.346 to 0.481, except for one sample (0.512) at 80 °C with maltodextrin. All values remained within microbiologically safe ranges for storage stability.

The desirability analysis identified the optimal processing conditions as 140 °C, pure maltodextrin, and R = 2.22 (Figure 2).

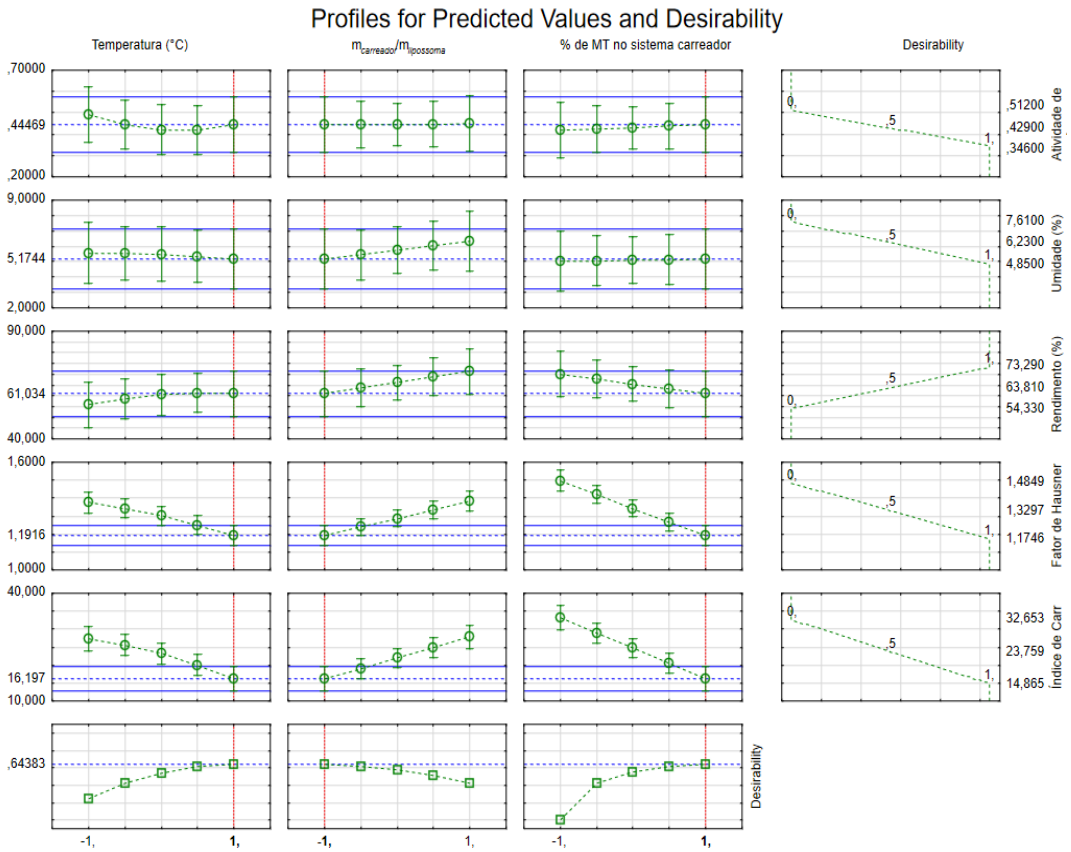

Figure 2. Profile of optimized values based on experimental results.

The incorporation of both empty liposomes (LB) and liposomes containing pequi oil (LOP) into the leave-in was achieved without compromising the stability of the formulations. All formulations remained homogeneous, with no signs of phase separation or precipitation throughout the 90-day observation period. The pH values remained within the range of 3.8 to 4.1.

The efficacy of the liposome formulations was evaluated to assess their performance in hair care applications. The tensile strength analysis showed significant differences between virgin and bleached hair tresses for both curly and straight hair ($p < 0.0001$), with straight hair demonstrating greater resistance to traction than curly hair, both before and after bleaching (Figure 3).

For curly hair, the non-bleached control (CH1) showed the highest tensile strength among curly samples, with a significant reduction observed after bleaching (CH2) ($p < 0.0001$). Treatment with all formulations (CH3 AT, CH4 AT, CH5 AT, and CH6 AT) did not significantly improve tensile strength compared to the bleached control, indicating limited mechanical recovery in curly hair.

In contrast, the non-bleached control of straight hair (SH1) also showed significantly higher tensile strength before bleaching process (SH2) ($p < 0.0001$). However, all treated straight hair groups (SH3 AT to SH6 AT) showed increased tensile strength relative to SH2, with statistical significance only observed for SH6 AT ($p < 0.01$), indicating a restorative effect of the formulation containing liposomes with pequi oil.

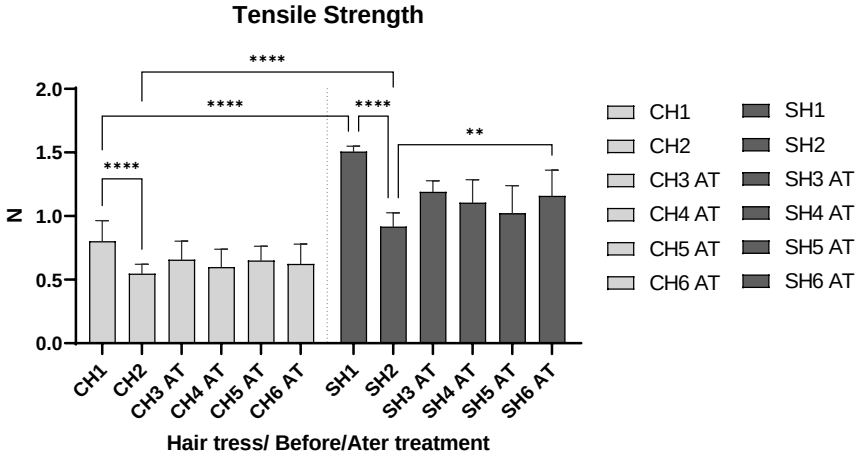

Figure 3. Tensile strength of curly (CH) and straight (SH) hair tresses before and after bleaching and treatment with different formulations. CH1 and SH1 represent non-bleached control tresses for curly and straight hair, respectively. CH2 and SH2 correspond to bleached controls. CH3–CH6 and SH3–SH6 refer to tresses treated after bleaching with: vehicle (AT), vehicle + free pequi oil, vehicle + empty liposomes, and vehicle + liposomes containing pequi oil, respectively. *Statistical significance: **p < 0.01; ****p < 0.0001.

The softness analysis showed a significant increase in measured values after bleaching for both hair types (Figure 4), reflecting reduced softness. In the curly hair group, CH2 exhibited significantly higher values than CH1 ($p < 0.05$), confirming the negative impact of bleaching on the hair texture. Most treated curly hair groups (CH3 AT, CH4 AT, and CH6 AT) showed a significant decrease in softness values compared to CH2 ($p < 0.05$, $p < 0.01$, and $p < 0.0001$, respectively), suggesting enhanced softness. CH4 AT and CH6 AT had values closest to CH1, indicating greater effectiveness.

For straight hair, bleaching significantly increased the values (SH1 vs. SH2, $p < 0.0001$). Treatment with SH3 AT, SH4 AT, and SH6 AT led to significant reductions in softness values ($p < 0.01$, $p < 0.01$, and $p < 0.0001$, respectively), indicating improved softness. In contrast, SH5 AT showed the highest softness value among the treated groups, suggesting that the empty liposome formulation reduced hair softness.

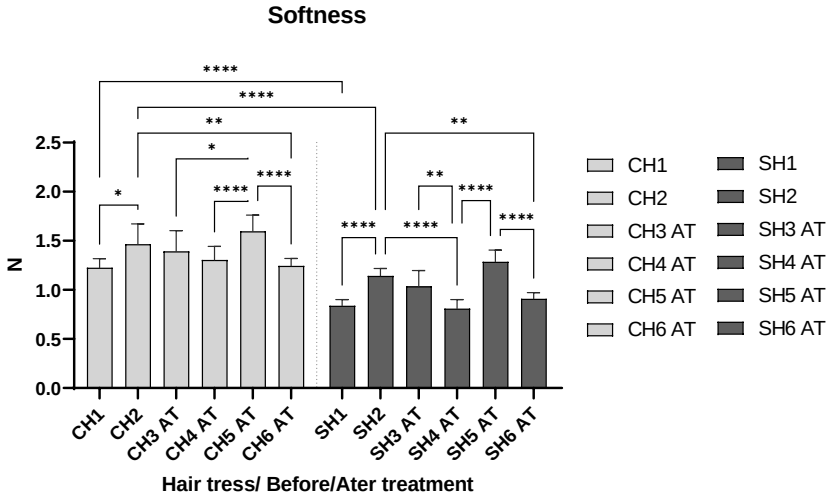

Figure 4. Softness of curly (CH) and straight (SH) hair tresses before and after bleaching and treatment with different formulations. CH1 and SH1 represent non-bleached control tresses for

curly and straight hair, respectively. CH2 and SH2 correspond to bleached controls. CH3–CH6 and SH3–SH6 refer to tresses treated after bleaching with: vehicle (AT), vehicle + free pequi oil, vehicle + empty liposomes, and vehicle + liposomes containing pequi oil, respectively. *Statistical significance: *p , 0.05; **p < 0.01; ****p < 0.0001.

4. Discussion

The increase in particle size for LPO can be attributed to the incorporation of the pequi oil in the lipid bilayer [2]. Despite this increase, the size remained in the nanometric range, appropriate for cosmetic applications [10]. The higher PdI in LOP suggests greater heterogeneity due to oil encapsulation, though values still present good homogeneity [11].

The yield and physical behavior of the spray-dried powders were influenced by temperature, carrier composition, and the R ratio. The use of maltodextrin performed better than Arabic gum at higher temperatures due to its lower hygroscopicity and higher glass transition temperature, improving process efficiency and powder stability [12,13].

Flow and compressibility data confirmed the relevance of maltodextrin for achieving better powder properties, while Arabic gum proved more effective at low temperatures due to its emulsifying capability and moisture retention. This complements the dual strategy of selecting carriers based on temperature-specific encapsulation goals [14]. Finally, the a_w values across all conditions remained within acceptable for dry cosmetic products, further supporting the optimized formulations.

The leave in formulation developed in this study exhibited a pH range of 3.8 to 4.1, which remained stable throughout the evaluation period. This pH is compatible with the isoelectric point of hair keratin, helping to maintain fiber integrity and minimizing cuticle swelling [15,16].

Chemical treatments such as bleaching are known to compromise the structural integrity of hair fibers, primarily through cuticle degradation and cortical damage, resulting in increased porosity and loss of mechanical resistance. These effects were confirmed in the present study, with both hair types showing a significant reduction in tensile strength after bleaching. Straight hair demonstrated greater resistance to traction than curly hair, both before and after bleaching, which is consistent with previous observations that straight hair has a more compact structure and less inherent fragility [1,17,18].

Although treatment with the tested formulations did not significantly improve tensile strength in curly hair, positive effects were observed in straight hair, particularly in the group treated with liposomes containing pequi oil. This result could be associated to nanoparticle size.

The size of nanocarriers plays a critical role in their ability to penetrate the hair shaft. Previous studies have demonstrated that nanoparticles around 600 nm are effective for this purpose, as hair movement can function like a mechanical pump, facilitating the entry of these particles into the follicles, where cuticles are approximately 600 nm thick [17,19,20]. In the present study, the liposomes had an average particle size below this value, suggesting their potential to penetrate not only the cuticle but also deeper layers of the hair fiber, such as the cortex.

The incorporation of pequi oil into liposomes resulted in improved tensile strength, particularly in straight hair, which may be attributed to the efficient delivery of bioactive components facilitated by the small particle size. This targeted penetration likely enhances the oil's ability to reinforce the hair fiber, promoting structural cohesion. Additionally, the improvement may be associated with the film-forming capacity of the liposomal system, which coats the hair fiber, enhancing surface smoothness and contributing to cuticle protection.

Softness analysis supported these findings. Lower force values after treatment, especially for CH6 and SH6, indicated increased softness, a desirable sensorial property in cosmetic applications. These outcomes are likely related to the emollient effect of pequi oil, as well as the ability of liposomes to form a uniform lipid film on the hair surface, which enhances cuticle alignment and smoothness [9].

Conditioning agents are known to reduce combing force [21]. However, the group treated with empty liposomes (SH5 AT) showed the highest softness value among the curly and straight hair samples, suggesting that the lipidic bilayer of the carrier itself may negatively impact the conditioning effects of the leave-in formulation.

In this context, these results reinforce the potential of liposomal encapsulation combined with spray drying as an effective delivery system for improving both mechanical and texture of chemically damaged hair. Furthermore, the differentiated responses between curly and straight hair emphasize the need to consider hair morphology when developing and testing cosmetic formulations.

5. Conclusion

The optimization of the spray drying process for liposomes containing pequi oil led to stable powders suitable for cosmetic applications. The most favorable conditions were identified through desirability analysis, demonstrating that higher temperatures and maltodextrin content enhance overall powder properties. When incorporated into a leave-in formulation, the optimized liposomal system improved the tensile strength and softness of bleached hair, with more pronounced effects observed in straight hair. These results reinforce the efficacy of liposomal delivery systems in enhancing the performance of natural active ingredients. Furthermore, the distinct responses between curly and straight hair showed the importance of considering hair morphology in the development of targeted hair care products. In summary, this study contributes to the advancement of innovative and effective raw materials, highlighting the potential of encapsulated pequi oil as a high-performance cosmetic ingredient derived from Brazilian biodiversity.

6. References

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