Novel delivery system for reducing water consumption by altering hair wash frequency

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Abstract

Background: Consumers are seeking opportunities to adapt their hair wash routines to incorporate more convenient and sustainable solutions, particularly to combat scalp oiliness.
Methods: For the delivery system, a combination of actives was used: Tea Tree, Sunflower, Rosemary and Pumpkin Seed. Clinical tests were performed using objective and subjective data (shampoo and conditioner, 1% active). Scalp oiliness was measured 1 and 7 days after using the product daily. A salon evaluation was conducted to determine extension of hair wash cycles.
Results: Nanoparticle suspension was successfully obtained. For oiliness reduction, panellists using the encapsulated actives presented a reduction of 47.4% and 61.2% after use encapsulated actives for 1 and 7 days. After seven days without washing their hair, panellists were asked about their ideal hair wash routine. The wash frequency of panellists using the placebo remained unchanged, while it has been reduced for the panellists using the nanoparticles regime (29.4%).
Conclusion: A new delivery system was developed intended to reduce hair wash cycles. Clinically, the proposed system can reduce scalp oiliness and alter the hair wash routine.

Keywords: lipid nanoparticle; encapsulation; delivery system; scalp oiliness; water saving; hair wash routine

Introduction.

Our hair care routines are evolving to meet the demands of the modern world, with consumers becoming more mindful of how their choices impact the world around them. Consumers are seeking opportunities to adapt their hair wash routines to incorporate more convenient and sustainable solutions, particularly those who have adopted intensive hair wash routines to combat scalp oiliness. With a growing consumer demand for time-saving solutions, coupled with an increasing awareness of the need to maintain scalp wellness and the impact scalp imbalances can have upon the hair, viable solutions to tackle oily scalps that will not compromise on the condition of the scalp or aesthetics of the hair is vital.

Lipid capsules are delivery systems based on solid lipids surrounding an oil core in which the active is dissolved. Cutaneous use of lipid nanoparticles presents several advantages, such as the chemical protection of the incorporated substances, allowing the skin application of labile molecules that are difficult to transport in traditional semi-solid formulations; improved active bioavailability, related to the possibility of modulating molecules release, promoting their skin penetration and retention. The latter has been explained by the lipid nanoparticles easiness to adhere to the Stratum Corneum, and the capability of allowing encapsulated molecules reaching the deeper skin layers, as described in literature. These properties are related to the lipid nanoparticles physiological lipid composition that can interact with the Stratum Corneum, creating its lipid rearrangement, which eases molecules penetration. Furthermore, published papers demonstrated that the nanoparticles small size also contributes to increase their adhesiveness and surface contact area, promoting the active influx through the skin.

There are just a few papers describing hair and scalp application of lipid nanoparticles. There are numerous advantages of encapsulation that could be obtained to hair/scalp use, including:

- Enhanced formulation stability and compatibility
- Active ingredients are protected from light, air, and other components of the formulation, resulting in increased active stability
- Controlled and targeted delivery of actives
- Slow release of actives allowing for enhanced penetration into the epidermis
- Provides occluding effect on the skin, reducing trans-epidermal water loss (TEWL) and thus maintaining skin hydration

Literature research also suggests the tendency of lipid capsules to diffuse in hair follicles. It is recognised as an important pathway for active delivery and intrafollicular penetration can be used to target specific structures. The penetration depth is thought to be linked to hair movement, which is mechanically driven and can be simulated by massage of the hair and scalp (i.e. applying products such as shampoo). It is also directly linked to the particle size of the lipid capsule. Capsules of 200 – 350nm are thought to be able to reach the region of the hair follicle in which the sebaceous glands are located, allowing the actives to operate where required to mitigate sebum production.

The aim of this study was to develop a novel encapsulation system to efficiently deliver actives to the hair and scalp and provide instant sebum reduction.

Due to their unique compositions, a selection of actives that could work synergically was co-encapsulated in a single lipid nanoparticle system. The combination of actives and their associated attributes are essential in maintaining homeostasis of the scalp and hair, reducing excessive sebum reduction, and preserving the integrity of the skin barrier. The selected actives were:

- Tea Tree Oil – largely consists of cyclic monoterpenes and sesquiterpenes, including terpinen-4-ol, α-terpinene, α-terpinolene and γ-terpinene
- Sunflower Oil – contains high levels of essential fatty acids, such as linoleic acid, linolenic acid, oleic acid, palmitic acid and stearic acid
- Rosemary Oil – contains a combination of triterpenes (including ursolic acid and oleanolic acid), tricyclic diterpenes (including carnosic acid and carnosol), phenolic acid and essential oils
- Pumpkin Seed Oil – contains a high content of polyunsaturated and saturated fatty acids, such as linoleic acid, linolenic acid, oleic acid, myristic acid and palmitic acid

Materials and Methods.

Delivery system development and characterization

The following INCI list was used to prepare the encapsulated product: Aqua (and) Melaleuca Alternifolia (Tea Tree) Leaf Oil (and) Sorbitan Stearate (and) Helianthus Annuus

(Sunflower) Seed Oil (and) Cetyl Palmitate (and) Lauryl Glucoside (and) Rosmarinus Officinalis (Rosemary) Extract (and) Cucurbita Pepo (Pumpkin) Seed Extract.

A lipid nanoparticle was prepared by mixing wall components and homogenizing them with an aqueous surfactant solution. A proprietary process was applied.

Macroscopic Aspect

Aspect, colour and viscosity were evaluated visually. Samples were analysed for the presence of phase separation, precipitates, heterogeneous aspects by checking in a clear glass without any instrument.

pH

The pH was measured directly in formulation (no dilution) using a calibrated (pH 4.0 and 7.0) potentiometer.

Particle size

Particle size was determined by laser diffraction technique. The formulation was directly added into the wet dispersion unit containing water under stirred until obscuration in the range of 2 - 8%. Refractive index used was 1.456. The results were expressed in $D_{V(50)}$, corresponding to particle diameters at the 50^{th} percentiles of the particle size distribution curve.

Release profile test

To evaluate the release profile, a sample of the delivery system (2 ml) was placed within a dialysis bag (12,000 Da cut, cellulose acetate), which was closed with clamps. The dialysis bag has a smaller pore size than the size of the delivery system capsules, thus allowing only solubilized actives to diffuse. The bag was placed into a release medium (ethanol-water 80:20, 32°C) which was agitated at a constant temperature. This agitation simulates general wear and erosion causing the contents (actives) of the encapsulate to be released and to pass through the pores of the dialysis bag, into the medium. Samples of the medium were collected and analysed by High Performance Liquid Chromatography (HPLC), to quantify Tea Tree Oil.

HPLC method consisted in C_{18} column (15 cm), mobile phase acetonitrile:water (80:20), mobile phase flow of 1 ml/min, detection wavelength of 274 nm and total run time of 30 min.

In vitro reconstructed human skin permeation

To demonstrate the delivery benefit of the delivery system onto the skin, an evaluation was performed using the Episkin™ model, which is an in vitro reconstructed human skin that is composed of the stratum corneum and the epidermis. The evaluation was used to simulate the level of skin permeation of actives compared to the non-encapsulated actives.

Samples of delivery system (100 µl) and the equivalent concentration of non-encapsulated actives were prepared and released onto the Episkin™ inserts which were placed above the release medium (500 µl). Samples of the release medium were collected for analysis after 1, 3 and 6 hours after application of encapsulated and the non-encapsulated actives. After 6 hours, the Episkin™ inserts were removed, cut and the actives extracted using solvent. All samples were analysed by High Performance Liquid Chromatography (HPLC), which confirmed the presence of Tea Tree Oil and thus the actives.

Clinical studies

Scalp Oiliness Evaluation

The instant improvement in scalp oiliness offered by the delivery system was evaluated both instrumentally and via sensory evaluation by an untrained panel of volunteers.

In order to determine the reduction in scalp oiliness, a Sebumeter evaluation was conducted where three different treatment regimes were evaluated:

- Shampoo and Conditioner containing encapsulated actives
- Shampoo and Conditioner containing un-encapsulated actives
- Shampoo and Conditioner containing no actives

The shampoo and conditioner formulations used in each treatment regime are shown below in Tables 1 and 2.

Ingredient	% w/w		
	Delivery system	Un-encapsulated actives	No actives
Sodium Laureth Sulphate	20.0	20.0	20.0
Cocamidopropyl Betaine	6.0	6.0	6.0
Cocamide DEA	3.5	3.5	3.5
Propylene Glycol	1.5	1.5	1.5
Delivery system	1.0	--	--
Cucurbita Pepo Seed Extract	--	0.01	--
Rosmarinus Officinalis Leaf Extract	--	0.025	--
Melaleuca Altenifolia Leaf Oil	--	0.04	--
Caprylyl Glycol	1.0	1.0	1.0
Water	To 100	To 100	To 100

Table 1: Shampoo formulations

Ingredient	% w/w		
	Delivery system	Un-encapsulated actives	No actives
Cetearyl Alcohol	0.7	0.7	0.7
Cetyl Alcohol	0.05	0.05	0.05
BHT	0.05	0.05	0.05
Dimethicone	2.0	2.0	2.0
Cetyl Trimethyl Ammonium Chloride	8.0	8.0	8.0
Delivery system	1.0	--	--
Cucurbita Pepo Seed Extract	--	0.01	--
Rosmarinus Officinalis Leaf Extract	--	0.025	--
Melaleuca Altenifolia Leaf Oil	--	0.04	--
Capryl Glycol	1.0	1.0	1.0
Water	To 100	To 100	To 100

Table 2: Conditioner formulations

20 panellists participated in each treatment regime, with both male and female participants within the age group of 18 – 65. The panellists were asked to refrain from washing their hair for two days prior to the evaluation, at which point their initial scalp oiliness (D0) was measured via Sebumeter analysis. The next day, the panellists washed their hair using the allocated regime and their scalp oiliness was measured again (D1 – first use). After seven

consecutive days of use, panellists had their scalp oiliness measured again (D7) and were asked to complete a questionnaire relating to their perceptions of their hair.

Hair Wash Cycle Evaluation

In addition to the scalp oiliness evaluations performed, an additional salon evaluation was conducted with a panel of untrained volunteers to determine consumer perception of their ability to extend their hair wash cycles following use of the delivery system.

The salon evaluation evaluated two different treatment regimes:

- Shampoo and Conditioner containing encapsulated active (delivery system) at 1% w/w
- Shampoo and Conditioner containing no actives (placebo treatment)

The shampoo and conditioner formulations used in each treatment regime were the same as the formulations used in the scalp oiliness evaluation, shown earlier in Tables 1 and 2.

20 panellists participated in each treatment regime, with female participants within the age group 20 – 57. The panellists were asked to refrain from washing their hair for two days prior to the evaluation, at which point they were asked to complete a questionnaire relating to their normal hair wash routines. The panellists were then asked to wash their hair using their allocated treatment regime (D0 – initial hair wash) before proceeding to wash their hair again at pre-determined time intervals:

- D3 – 2 day wash cycle
- D7 – 3 day wash cycle
- D13 – 5 day wash cycle
- D20 – 7 day wash cycle

At each interval, the panellists were asked to complete a questionnaire to determine whether they had wanted to wash their hair and whether they felt that they could have wait longer before washing their hair. Once the study was complete, the panellists completed a final questionnaire to determine their ideal hair wash routine following use of their allocated treatment regime, which allowed an average change in wash frequency to be determined.

Results.

Just after preparation, samples were homogeneous, stable, without visible phase separation, colour was white, and no visible particles were seen in the suspension. The resulting final pH of the formulations was 10.6 ± 0.6. Average DV(50) was 0.19 ± 0.01 µm.

In vitro release study and permeation

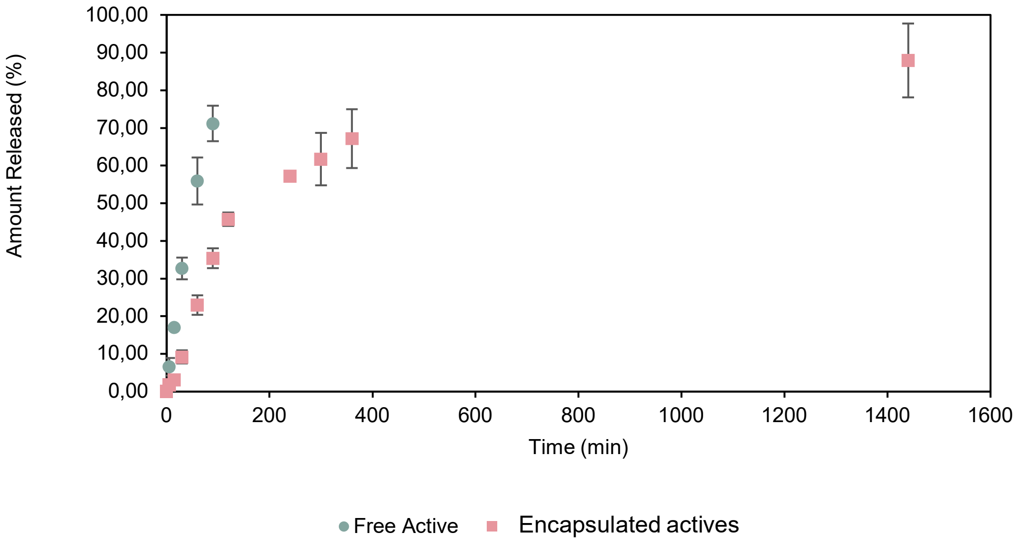

Figure 1: The release profile of encapsulated actives compared to the free actives (tea tree oil quantification)

As demonstrated in Figure 1, the actives contained within the delivery system capsule were released at a slower rate compared to the free actives. This suggests that the release of the actives is more controlled from the encapsulate, thus demonstrating the benefits of encapsulation and its ability to deliver continued benefits.

Release mechanism was assessed applying Korsmeyer-Peppas equation to the experimental data. Release half-life was calculated by first order release equation.

Free active diffused with half live of 58 min. Encapsulated actives released from the particles by anomalous transport that is the association of diffusion and particle erosion. The index from Korsmeyer-Peppas equation was 0.61. Half-live of release was 136 min. This result indicates that the release is controlled by the encapsulation and the rate is reduced compared to the free form.

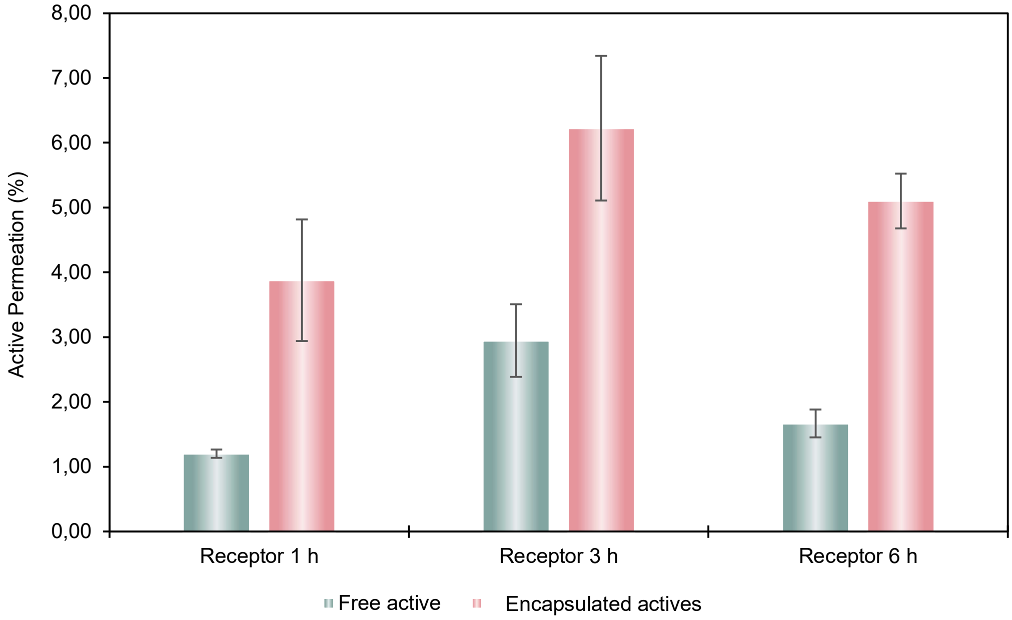

Figure 2: Skin permeation using Episkin^{TM} model

The increased permeation of the encapsulated actives compared to the free actives, as shown in Figure 2, demonstrates the advantage encapsulation provides to the delivery system in terms of the effective delivery of actives to the skin.

Clinical evaluation

The results of the Sebumeter evaluations are presented in Figure 3.

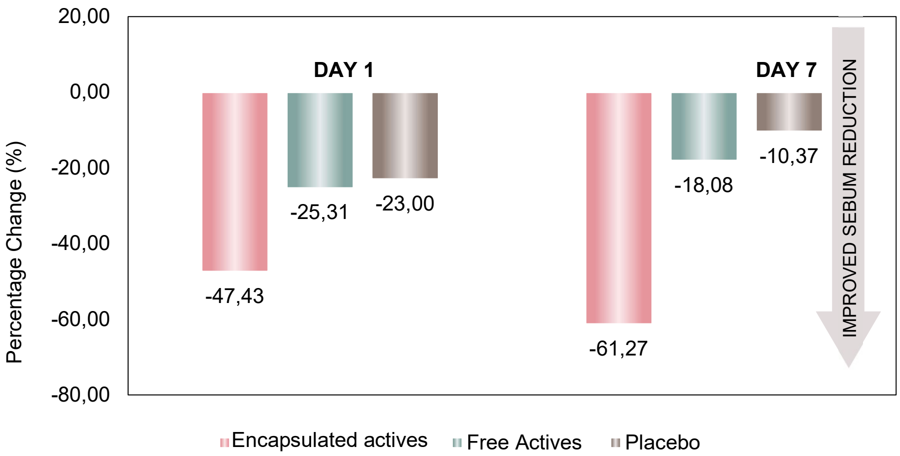

Figure 3: Sebumeter evaluation results

As demonstrated in Figure 3, the encapsulated actives delivered a significant reduction in scalp oiliness, which was effective from the initial application and continued to improve with repeated use. This result was consumer perceivable compared to the other regimes tested.
Following the final Sebumeter reading (D7), the panellists completed a questionnaire where they were asked to rank parameters relating to scalp oiliness and clean hair sensation on a scale of 1 – 5, with 1 being much better and 5 being much worse.

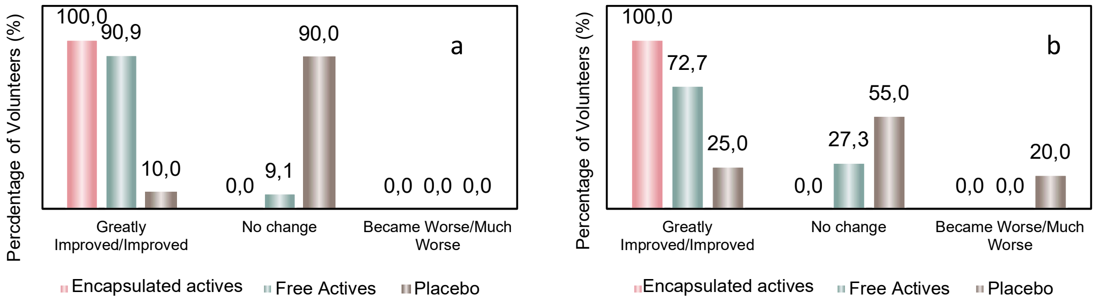

Figure 4: Volunteer perception of scalp oiliness after the first treatment (a) and after completing the full seven day treatment regime (b).

Figure 4a shows that panellists noticed a distinct reduction in the amount of sebum produced on the scalp from the first treatment, with 100% of panellists indicating an improvement in the feel of their scalp following use of the encapsulated actives after completing the full treatment regime, as demonstrated below in Figure 4b.
Sensory evaluations also demonstrated an improvement in the hair look and feel following treatment with the encapsulated actives, compared to the hairs' state following their usual hair wash regime.

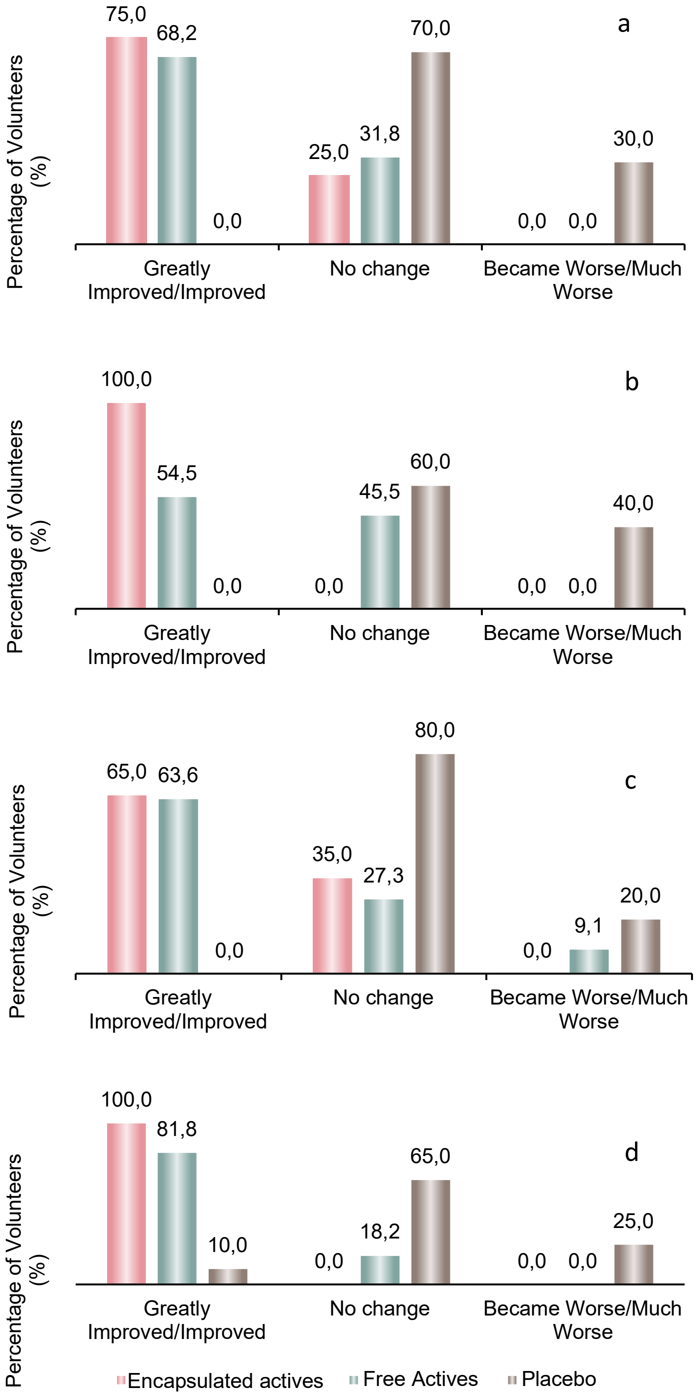

Figure 5: Volunteer perception of hair shine (a), hair smoothness (b), hair dry feel (c) and clean hair sensation (d) after completing the full treatment regime

As demonstrated in Figure 5a, all panellists indicated that hair shine was not detrimentally impacted following use of the encapsulated actives, with ¾ of panellists indicating that hair shine was either improved or greatly improved.

As shown in Figure 5b, 100% of panellists indicated that hair smoothness was either improved or greatly improved following use of encapsulated actives.

As demonstrated in Figure 5c, the panellists indicated that hair dry feel was not detrimentally impacted following use of encapsulated actives, with the majority of panellists indicating that hair dry feel was either improved or greatly improved

The panellists were also asked whether they experienced a clean hair sensation, of which 100% of panellists indicated improvements in the feel of clean hair following the repeated use of encapsulated actives, as is shown in Figure 5d.

Hair wash regime evaluation

After two days of no hair washing, 100% of panellists for both treatment regimes indicated that they did not want to wash their hair, with 100% of panellists using the encapsulated actives regime indicating that they could wait longer before washing again, as demonstrated in Figure 6a. 95% of the placebo group indicated that they could wait longer before washing their hair, with one panellist commenting that their hair already felt oily.

After three days of no hair washing, 70% of panellists using the encapsulated actives regime indicated that they did not feel the need to wash their hair, compared to 20% of panellists using the placebo regime, demonstrated below in Figure 6b. This is further supported by the results demonstrated in Figure 6b, where 85% of panellists using the encapsulated actives regime indicated that they could wait longer before washing their hair, compared to 25% of panellists using the placebo regime.

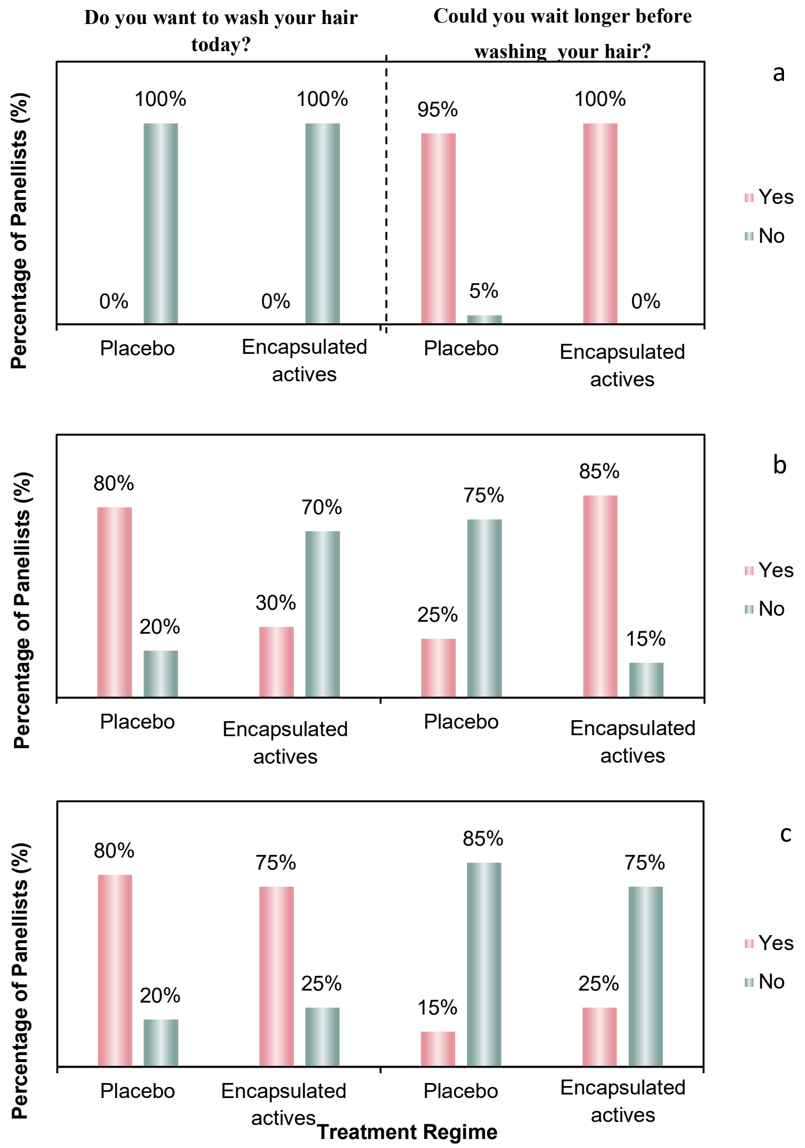

Figure 6: Panellists' perception after two days of no hair washing (D3) (a), three days of no hair washing (D7) (b) and five days of no hair washing (D13) (c).

Figure 6c demonstrates that after five days of no hair washing, 75% of panellists using the encapsulated actives regime felt that they needed to wash their hair, with 80% of panellists using the placebo regime agreeing. The results also demonstrate that 75% of panellists using the encapsulated actives regime indicated that they could not wait longer to wash their hair, with 85% of panellists using the placebo regime agreeing.

The evaluation concluded after D20 (seven days of no hair washing) and panellists were asked to complete a questionnaire about their ideal hair wash routine following use of their allocated treatment regime.

Based on their answers, the change in wash frequency for each treatment regime was determined and the results are presented below in Figures 7 and 8.

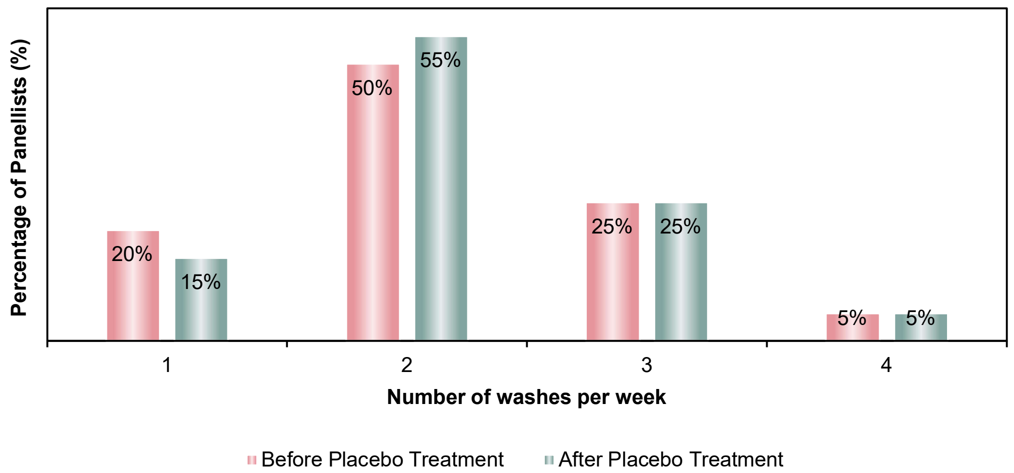

Figure 7: Change in hair wash frequency following use of the placebo regime

Figure 7 demonstrates that the wash frequency of panellists using the placebo regime largely remained unchanged, with one panellist who originally indicated that they usually wash their hair once per week now feels that they need to wash their hair twice per week.

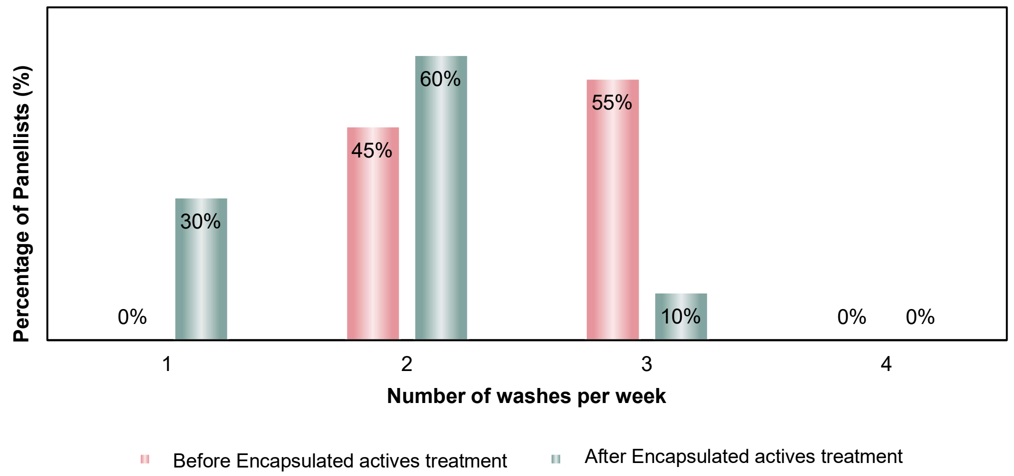

Figure 8: Change in hair wash frequency following use of the Nutrinvent Balance regime

Figure 8 demonstrates that the wash frequency of panellists using the encapsulated actives regime reduced, with a distinct reduction observed for three washes per week, with more panellists indicating that they could wash their hair either once or twice per week.

The results indicate an average reduction in wash cycles of 29.4% for the encapsulated actives regime, compared to an increase of 2.2% for the placebo regime.

Discussion.

A lipid nanoparticle was successfully produced. All actives could be co-encapsulated in an innovative capsule allowing stability and performance improvements. Particle size was nanometric, with narrow polydispersity. All characterizations showed and adequate system for cosmetic application.

Regarding release profile, mechanism was calculated as a combination of diffusion and erosion. Diffusion will occur naturally between areas of high and low concentrations. When the delivery system is applied to the scalp, a concentration gradient is formed between the scalp and the capsules, at which point the diffusion process begins.

Erosion will also occur upon application, where the outer layer of the capsules will start to degrade and the actives are released. Diffusion and erosion will occur simultaneously, which accelerates the release of actives. The rate of diffusion will eventually decrease as the concentration gradient reduces and the actives will only be released by erosion.

This process demonstrates the ability for the capsules to deliver the actives to the scalp for a prolonged period of time (i.e. the actives do not remain encased).

Using Episkin model, it was possible to demonstrate increase in skin permeation after the use of the encapsulated actives. In all time points, concentration found in release medium was higher when using the delivery system. This difference in permeation, combined to the slow release can explain the improvement of the clinical activity obtained in panellists' tests.

Regarding immediate oiliness reduction, the delivery system applied to shampoo and conditioner was able to significantly reduce the scalp oiliness after one use. The continuous use for 7 days increased the efficacy, in opposite to the results obtained using placebo or free actives, where the oiliness was reduced in the same proportion. The objective measure of scalp oiliness was corroborated with sensorial results, indicating that volunteers with higher improvement of oiliness were the ones that used the encapsulated actives.

The delivery system presented another advantage compared to conventional shampoos for oily hair that only have increased surfactant concentration. The encapsulated actives treatment also increased sensorial properties and did not have any detrimental effect on shine, smoothness, and dry feel, usually reported when regular shampoos are used.

The salon test for the evaluation of hair wash regime showed difference between placebo and treatment containing the delivery system after an interval of 3 days between washes. At this time interval, panellists using placebo treatment wanted to wash their hair and couldn't wait longer before washing (80 and 75%, respectively) whereas the ones that used the treatment with encapsulated actives were still comfortable in wating more days before washing (85%). This result reflects the data for the ideal wash cycle routine. For the volunteers that used the placebo treatment, there was no willing in changing their routine. A different situation was seen when the panellists used the encapsulated actives. Volunteers that washed their hair 2 or 3 times a week could change their routine to 1 or 2 times a week, showing almost 30% in reduction.

Using data from Brazil in terms of shower and hair wash routines, it was calculated the sustainability benefits of the change in the routine seen by the panellists using the encapsulated actives shampoo and conditioner.

One hair wash (shampoo and conditioner) uses an average of 88 l of water and takes 8 min. Calculating the difference of water use between initial and final hair wash routines proposed by the panellists, for every tonne of encapsulated active used, it is estimated to save 129.4 million litres of water. The carbon emissions related to heated water that can be avoided are 229,412 kgCO_2.

By accepting to wash less frequently their hair, consumers can save water equivalent to the annual equivalent drinking water for 155,982 people, avoiding carbon emissions equivalent to 4,588 return flights from Paris to London. One tonne of encapsulated actives can make 400,000 bottles (250 ml) of shampoo or conditioner.

By proposing a new benefit for the delivery system developed in this project, it was possible to re-evaluate the hair wash routine, with positive and direct impact on the planet, and on the daily life (reduced time in shower), maintaining hair and scalp clean, with adequate sensorial for more days.

Conclusion. A novel delivery system was developed combining 4 actives in a lipid system. Release profile and permeation showed slow and continuous delivery with higher permeation than free actives.
The delivery system was added to shampoo and conditioner at 1% w/w and tested clinically. Immediate oiliness reduction was significantly higher than placebo and free actives after one and 7 days of use. Sensorial properties of hair were improved.
Once using the treatment with encapsulated actives, hair wash routine could be changed by reducing the hair washes by 29.4%. This reduction in hair washes/week led to water saving and carbon emission reduction that positively impact our environment. This was the first time that a cosmetic active offers the benefit for extending interval of hair washes, keeping hair with good sensorial.

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Conflict of Interest Statement.
NONE.

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