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CHALLENGES FOR FORMULATING NATURAL COSMETICS: COMPARATIVE PHYSICOCHEMICAL STUDIES ON NATRUAL AND SYNTHETIC MADE SHAMPOO

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ABSTRACT

There has been a gradual increase in trends towards preparations of natural cosmetics due to its impressive efficacy, sustainability, non-toxicity, and feel of living with nature. Different words were being used by the cosmetic industry in label claims as Natural, Organic, Herbal, Vegetable, and Green ingredients. These magical words create a mass of confusion among the customers to choose the right product. This article deals with preparation of natural shampoo and its comparative physicochemical evaluation with synthetic made shampoo. It also discusses the challenges faced by the formulators/manufacturers to formulate a completely natural shampoo.

KEYWORDS: **Shampoo, Eco-cert, Physicochemical evaluation, Natural cosmetics**

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INTRODUCTION

From the perspectives of end user, the purpose of the shampoo as follows - 1. To remove sebum, dandruff and atmospheric pollutants from the hair and scalp[1]. 2. To remove the residues of previously applied hair treatments, e.g. polymeric constituents from styling lotions and hair sprays. 3. It is expected to deliver an optimum level of foam to satisfy the expectation of the user. 4. To leave the hair in a satisfactory condition after rinsing so that it can be combed easily both in the wet and dry state. 5. To anticipate performing as a vehicle for the deposition of beneficial materials onto the hair and scalp. 6. To be non-toxic and non-irritating to the hair and the scalp. 7. To be non-damaging to the tissues of the eye if inadvertently splashed.

Regulations of USDA and Eco-cert

In order to make a natural/organic shampoo, it is important to understand the regulations laid by the regulatory bodies[2]. According to U.S Department of Agriculture's (USDA) National Organic Program (NOP), the term organic may be used on product labels when certain conditions are met[2]. It is further classified four types of label claims namely

1. "100% organic"
2. "Certified organic" requires 95% organic content
3. "Made with organic" requires 70% organic content.
4. "Contains organic" This label claim requires less than 70% of certified organic ingredients/contents in a product and also cannot bear the USDA Organic seal

The term "Organic" and "Natural" are not defined in either of these laws or the regulations that FDA enforces under their authority[2]. The product claim such as natural or herbal or vegetable or green is due to derivative of the ingredients from plant source. Another famous certification body is Ecocert; it is one of the famous inspection and certification body established in France in 1991 by agronomists aware of the need to develop environmentally friendly agriculture. It is specialized in the certification of organic agricultural products. This program attempts to apply a food law to cosmetics. Ecocert therefore checks the absence of GMOs (Genetically Modified Organisms), parabens, phenoxyethanol, nanoparticles, silicon, PEG (Polyethylene Glycol), synthetic perfumes and dyes, animal-derived ingredients (unless naturally produced by them: milk, honey, etc.). It also checks the biodegradable or recyclable nature of packaging. Ecocert Organic/Natural Standard imposes that a minimum of 95% of the total ingredients come from natural origin. Many of the cosmetic products been labelled as an Eco-cert in India. In a similar fashion, there are many certifications bodies like BDIH (Germany), COSMOS (EU) and OASIS (USA). This paper avoids dealing with various regulatory bodies and its requirement to claim Natural/Organic cosmetics, which in itself a large subject. Based on above regulations and requirement, formulated a formulation of Eco-cert organic shampoo.

Formulation of Ecocert Organic Shampoo

INCI (International Nomenclature for Cosmetic Ingredients)	% w/w
Aqua	Upto 100
Disodium Cocyl Glutamate	10.00
Decyl Glycoside	5.00
Sapindus Mukorossi fruit extract	2.00
Hydroxyethyl Cellulose	1.00
Aloe Barbadensis (Aloe) Leaf Juice	2.00
Lavandula Angustifolia (Lavender) Flower Water	1.00
Hibiscus Rosa Sinensis Leaf Extract	1.00
Glycerin (and) Leuconostoc/Radish Root Ferment Filtrate (and) Lonicera Japonica (Honeysuckle) Flower Extract (and) Lonicera Caprifolium (Honeysuckle) Extract (and) Populus Tremuloides Bark Extract (and) Gluconolactone	1.50

MATERIALS AND METHODS[3-8]

Preparation of shampoo

1. Disperse and slow addition of Hydroxyethyl Cellulose into water (Aqua) using a propeller stirrer having 1200-1500rpm to form a gel. 2. Pre-mix the surfactants blends like Disodium Cocyl Glutamate, Decyl Glycoside and Sapindus Mukorossi fruit extract, added into the above bulk with gradual mixing. 3. Followed by the addition of Aloe Barbadensis (Aloe) Leaf Juice, and Hibiscus Rosa Sinensis Leaf Extract. 4. Finally added the components "Glycerin (and) Leuconostoc/Radish Root Ferment Filtrate (and) Lonicera Japonica (Honeysuckle) Flower Extract (and) Lonicera Caprifolium (Honeysuckle) Extract (and) Populus Tremuloides Bark Extract (and) Gluconolactone", and mixed well.

Colour

This scale is often referred to as Pt-Co, Platinum-Cobalt, Hazen or APHA Colour. All terms are interchangeable and equally valid. It is used to measure clear to dark amber liquids. The scale was originally defined by specified dilutions of a platinum-cobalt stock solution, ranging from 0 at the light end of the scale to 500 at the darkest. The scale is now available in a digital form at on Automatic instruments. The scale is used extensively in the water industry but also for clear oils, chemicals and petrochemicals such as glycerine, plasticisers, solvents, carbon tetrachloride and petroleum spirits. Using this technique, measured the colour of the shampoo and reported in Table1

Odour

The organoleptic evaluation was conducted by panel members to perceive odour of the sample. The observations were recorded in Table1.

pH

10% solution of the shampoo was diluted using distilled water. The prepared sample was checked for pH using a digital pH meter at 25^0C

Solid Content

Moisture Determination Balances provide a primary method for accurate moisture measurement by using the loss on drying (LOD) method. The shampoo is placed in the sample pan. The integrated balance weighs the initial sample. The balance periodically weighs the sample and when the test completed, automatically calculates the moisture content (or solid content) of the initial sample. % Solid content was measured at 105 deg.

% Active Detergent

The active detergent (%) - Anionic, Cationic and Non-ionic surfactants were quantified by using Metrohm titrimetric method.

Surface Tension

Surface tension of air-water interface was measured by using Du Noüy ring method. It is most widely-used methods for measuring the surface tension due to rapid, simple and does not need to be calibrated using solutions of known surface tension. This instrument has an accurate micro-balance and a precise mechanism to vertically move the sample liquid

in a glass beaker. It has a computer-controlled arrangement that can move the table holding the liquid very slowly (~100 m/s). Average of triplicate values were recorded in Table 1.

Detergency

Ideally, shampoo used to remove the oils, dirt, skin particles, dandruff, environmental pollutants and other contaminant particles that gradually build up in hair. Detergent ability of product is the cleansing property which is calculated by sebum removal method from the hair. 1% solution of shampoo solutions were evaluated using gravimetric method by Thompson et al. protocol.

Viscosity

The viscosity of the shampoo was determined by using Brookfield Viscometer DV-II+ PRO Digital Viscometer. The viscosity of shampoo was measured at 25^0C at 20 RPM. The reported value was average of triplicate.

Foam Height

Foam has an aesthetic utility in many detergent and personal care products. Foaming properties such as speed, quality, quantity and longevity are the key attributes for many cleansing product. The foam height of 2% solutions was measured. Measurements were taken using Ross Miles apparatus as per BIS specification.

Foam Stability

Foam stability is an important parameter in cleansing formulations. The rate of weakening defines the stability of the foam. Foam with R50 values higher than 50% can be regarded as metastable. Foam stability was measured after 10 minutes.

Zein dissolution method (Mildness)

Mildness was evaluated by measuring dissolution of corn protein 'Zein'. The Zein solubility test method can be used to investigate the irritation potential (harshness) of a surfactant-based product on the skin. The Zein solubility test method is an in vitro technique based on the solubilisation of water insoluble corn protein. The increasing solubility of Zein by a surfactant can be correlated to skin irritancy. The test result was done triplicate using 1% solution of shampoo.

Stability Studies

Stability studies were performed in accordance with ICH guidelines for accelerated testing with required modifications. The shampoo was taken and kept at room temperature (25 ± 2^0C) as well as refrigerator (4±2^0C) for duration of one month. The samples were tested for their physical appearance, pH, viscosity, % detergency and foam stability. In addition to that samples were monitored at 45^0C and 45^0C + 75 RH.

Performance comparison

To evaluate the overall performance of Eco-cert shampoo in comparison with synthetic made commercial shampoo, 10 volunteers were asked to blind test two shampoos formulations, one containing Eco-cert shampoo and another containing synthetic made commercial shampoo. They were asked to evaluate and score the formulations for colour/odour, appearance, speed of foam, amount of foam, foam stability, usage feel, after feel, ease of washing, dryness and overall liking. Figure 1 shows the average scores reported for each attribute.

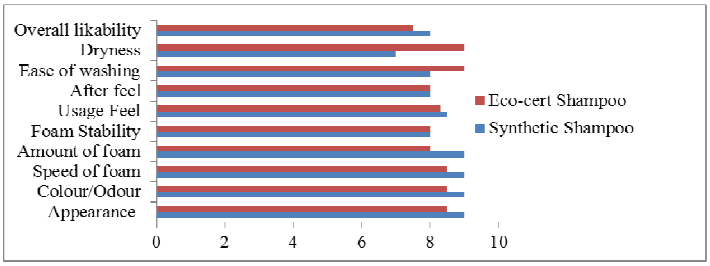

Figure 1
Performance comparison of Eco-cert and synthetic shampoo

Preservative efficacy test

Natural Ingredients are susceptible to microbial contamination. To check whether the products (Eco-cert shampoo) having the micro-organisms falls within the regulated limited and compared against commercial shampoo, hence performed preservative challenge test (USP 51). This test challenges preserved products by inoculating the formulation with a variety of microorganisms, mould and fungus. Microbiological testing is performed at 7, 14, 21 and 28 day intervals. The inoculum concentration is relatively high to provide an indication of how the preservative performs under "real life" conditions. To pass USP 51, bacteria must show ≥2.0 log reduction from the initial count at 14 days, and no growth in colony forming units (CFU's) through the 28 day count as shown in figure 2. For yeast & mould there should be no increase from the initial calculated count at 14 days through the 28 day count. Both the products are well preserved as per USP 51.

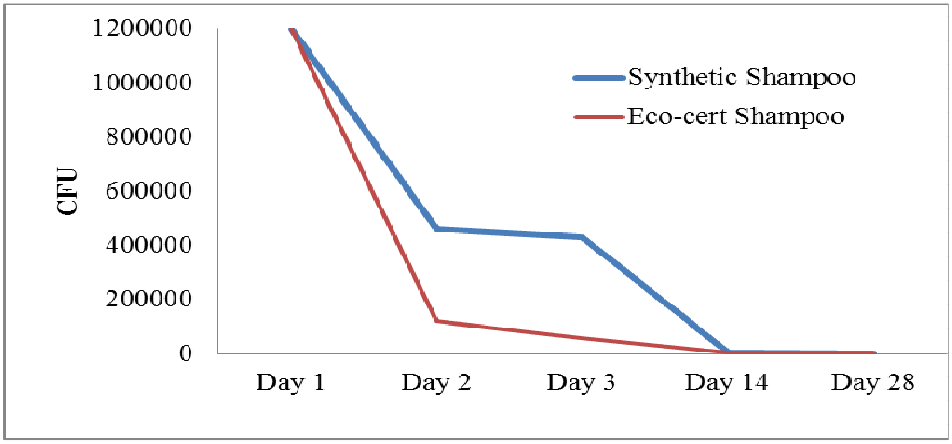

Figure 2
Preservative efficacy for Eco-cert and synthetic Shampoo

Table 1
Physiochemical properties of shampoo

Parameters	Ecocert organic Shampoo	Synthetic Marketed Shampoo
Appearance	Amber colour Liquid	Colourless
Colour*	350	30
Odour	Characteristic Herbal	Characteristic
Solid Content (%)	25.3	26.5
Active Detergent (%)	12.0	11.5
Surface Tension (dynes/cm)	32	34
Detergency (%)	93	95
Viscosity (cP)	2575	3010
Foam Height (ml)	165	169
Foam Stability (ml)	162	165
Zein dissolution (%)	20 ± 2.3	82 ± 3.2
Stability study	Stable	Stable
Preservative Efficacy Test	Passed	Passed

**** 0 at the light end of the scale to 500 at the darkest***

RESULTS AND DISCUSSION

As seen from the test results, all the parameters were found to be comparable with commercially available product except colour, odour, appearance and Speed/amount of foam. However, the foam volume was at par using Ross-Miles column but the sensorial feels were different. Ecocert organic shampoo perceived loose lacy foam structure whereas the synthetic marketed shampoo forms a dense lather. It is an ardent task to compete with synthetic foaming agent especially the sensorial attributes like speed and feel of the foam. In some cases during sensorial evaluation, the amount of foam

is perceived less over synthetic made shampoo. Dryness and ease of wash are significantly better for Eco-cert shampoo. There is a false notion among the customers that shampoo that 'foams well works well'. It is noteworthy to mention that the purpose of shampoo is to cleanse and remove the dirt, excess sebum, dandruff, environmental pollutants and other contaminant particles without damage the skin and hair proteins. But the synthetic detergent foams well and feel better but damage the skin and hair protein as evident from Zein dissolution test method.

CONCLUSION

It is evident that a lot of challenges for formulators to mimic the aesthetics (colour, odour) and performance characteristics (sensorial attributes) of synthetic ingredients with natural/organic alternatives by keep an eye on regulation, consistent sourcing and costing etc. However, the need of an hour is customer must be educated if 100% organic (Natural) cosmetic ingredients used in the formulation, the aesthetic may not be comparable to synthetic made formulations. By considering other factors like mildness, safety, toxicity, and sustainability there is a strong need to change the customer's mind-set and the onus lies on cosmetic manufacturers and formulators. At the moment, there are limited thickening agents available for natural formulators. Moreover, strong research is needed to understand the interaction between naturally derived surfactant-thickener system. In formulation, surfactant-thickener system plays an important role in amount/speed/feel/cleansing efficacy of the product. The study aid the formulator not only to understand the molecular level but also to formulate and mimic the sensorial feel of synthetically derived product.

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