

Formulation and Evaluation of Anti Dandruff Solid Shampoo

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Abstract: Shampoo is one of the most popular beauty products that clean the hair. Modern shampoo formulations go far beyond the simple cleaning of hair. Additional benefits are expected, for example, conditioning, smoothing of the hair surface, good health of hair, i. e., hair free of dandruff, dirt, grease, and lice, and, above all, hair that is safe to use. This research involves the development of a solid shampoo using different antidandruff agents in order to respond to the current demands of consumers. The formulation containing salicylic acid was chosen as the best formulation among the six batches. Tests were conducted to determine the requirements for developing a product capable of being able to wash and moisturize while remaining solid before washing. Among the product quality tests, microbiological analysis and dry weight determinations were conducted to calculate the amount of water and volatiles in the shampoo and check for microbial contamination. Using a solid shampoo that is sulphate-free, silicone-free, and plastic-free, a quick and improved way of cleaning was obtained with creamy foam that is abundant and sustainable. Packaging designs were created and recycled paper was used for packaging. In addition, stability tests were conducted for a period of one month and the formulation was found to be stable across all parameters.

Keywords: Solid shampoo, Antidandruff activity, Salicylic acid, *Malassezia* species

1. Introduction

A shampoo is a preparation containing surfactant in a suitable form – liquid, solid, or powder which when used under the specified conditions will remove surface grease, dirt, and skin debris from the hair shaft and scalp without adversely affecting the user. Most shampoos contain water, a detergent (cleaning agent), surfactant (lather making agent), salt, fragrance (natural and artificial), preservative, and food colouring. With the exception of water and salt (sodium chloride), different chemical compounds are used depending on the desired result of the shampoo. ⁽¹⁾

Dandruff is a chronic scalp condition that causes scaling, itching and redness of the scalp by shedding epidermal cells. The cause of dandruff is a fungus called *Malassezia restricta* and *Malassezia globosa*. *Malassezia*, formerly known as *Pityrosporum*, is a yeast that causes skin and scalp infections. For the treatment of dandruff, hair care preparations typically contain antifungal agents. Many of these products have side effects such as loss of hair, increased scaling, itching, irritation, nausea, and headaches. Dandruff can be treated in two ways, using herbal-based antidandruff shampoo and using chemical-based antidandruff shampoo. The dandruff shampoo bar was developed to be effective in treating the problem ⁽²⁾ The choice of "active" depends on the regulatory requirements in the market to be served. Currently, there are five ingredients approved for use in over-the-counter (OTC) anti-dandruff products in the United States. Zinc pyrithione and selenium sulfide are the most popular active ingredients, followed by salicylic acid, sulfur, and coal-tar solution. Except for coal tar and salicylic acid, the other three ingredients are used as suspensions in shampoo formulation. Many shampoos also contain vitamins and moisturizing alcohols to prevent too much of the hair and scalp's natural oils from being stripped away during cleansing. The purpose of antidandruff agents is to reduce the formation of dandruff flakes ⁽³⁾ The aim of

this study was to design a solid shampoo for oily, normal and dry/damaged hair that lasts longer than a traditional liquid shampoo, is eco-friendly and contains no sulphates, plastics, silicones or other additives that would extensively strip the natural oils from the hair ^(4, 5)

2. Literature Survey

Several researches were developed for the formulation of powder shampoo using Medicinal plants such as Onion Powder, Rose Petal, Lemon Grass, Flaxseed or Linseed, Hirda, Bahera, Black tea, Brahmi, Triphala, Bhringraj, Ginger Root, Ashwagadha, Shikakai, Feenu greek, Shatavari, Heena, Wala, Aloe vera Powder, Nirgudi Powder, Bavachi, Jatha mansi, Tulsi, Neem, Hibiscus Flower, and Retha. The various quality control parameters were checked. The latest research has performed that Bhringraj Powder exhibits excellent antifungal behaviour. The antidandruff shampoo was formulated by adding Bhringraj Powder as the principal ingredients of the antidandruff. As additives play an important role in determining any formulation's efficiency, stability and aesthetic appeal, this point was held in mind when selecting the additives for shampoo formulation. .

3. Methodology

Materials

Salicylic acid and sulfur are used as anti-dandruff agents. Sodium cocoyl isethionate and Cocamido propyl betaine was used as surfactants. All the chemicals were purchased from Yarrow Chem Products, Mumbai. ⁽⁵⁾

Methods Preparation of Anti-dandruff shampoo bar

Anti-dandruff shampoo bar was prepared by the formula given in table 1. Six formulations were prepared by using two anti-dandruff agents at various concentrations. Phase 1 ingredients (Castor oil, Lavender oil and Cocoa butter) are

heated in a container, one by one until it is clear and homogenous. In a separate container added phase 2 ingredients (Sodium cocoyl isethionate, Cocamido propyl betaine, Salicylic acid/Sulfur, Kaolin and Sodium benzoate) and mixed well. The mixture was placed in a double boiler to prevent clumps. Slowly added phase 1 to phase 2. Cooled the mixture for sometimes. After cooling, added essential oils and preservative. After it had reached play dough consistency, then gently pressed into a mold. Allowed to solidify at room temperature for 24 hours and unmolded. ^(6, 7)

Table 1: Formula for Anti-dandruff shampoo bar

S. no	Ingredients	(50g)	F1	F2	F3	F4	F5	F6
1	Sodium cocoyl isethionate	28	28	28	28	28	28	28
2	Cocamido propyl betaine	8.5	8.5	8.5	8.5	8.5	8.5	8.5
3	Salicylic acid	3	3	2.5	2	—	—	—
4	Sulfur	3	—	—	—	3	2.5	2
5	Cocoa butter	3	3	3	3	3	3	3
6	Kaolin	3	3	3	3	3	3	3
7	Castor oil	3	3	3	3	3	3	3
8	Lavender oil	1	1	1	1	1	1	1
9	Sodium benzoate	0.50	0.50	0.50	0.50	0.50	0.50	0.50

Evaluation of Anti-dandruff Shampoo bar

Physical appearance and Homogeneity ^(8, 9)

The prepared formulations were evaluated in terms of their clarity, foam producing ability, fluidity and homogeneity.

Foaming ability and foam stability ^(10, 11)

50ml of the 1% shampoo bar solution was placed right into a 250ml graduated cylinder and secured the cylinder with hand and shaken for 10 times. The overall volumes of the foam contents after 1 minute shaking were recorded. The foam volume turned into calculated handiest straight away after shaking the extent of foam at 1-minute periods for four minutes were recorded.

Dirt dispersion ⁽¹²⁾

Two drops of 1% each shampoo bar was added in a large test tube contain 10ml distilled water. 1 drop of Indian ink was added. The test tube has stopper and shaken for 10 minutes. the amount of Ink in the foam was estimated as none, light, moderate or heavy.

Percentage solid content ⁽¹³⁾

The percentage solid content was determined by weighing about 4g of shampoo bar in evaporating dish. The weight of the dish and shampoo was determined. The liquid portion of shampoo was evaporated by placing in heating mantle. Finally, the weight of solid contents after complete drying present in shampoo bar was calculated.

Determination of pH ^(14, 15)

The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C.

Moisture content ^(16, 17)

The moisture content was used to estimate the % of water in the shampoo bar by drying the shampoo bar to a constant

weight. The bar was weighed and recorded as wet weight of sample.

And was dried from 100 to 115°C using a dryer. The sample was cooled and weighed to find the dry weight of sample.

The moisture content was determined by using the formula

$$\% \text{ Moisture content} = \frac{\text{Initial weight} - \text{final weight}}{\text{Final weight}} \times 100$$

Anti-bacterial activity ⁽¹⁸⁾

The screening was done by cup plate method. The formulations were tested against *Bacillus subtilis*. A loopful of the pure bacterial culture was suspended in nutrient broth and incubated for 24 hours. Nutrient agar media was sterilized and poured into plates. After solidification, 0.1ml of the inoculum was spread over the agar evenly using L rod. A sterile cork borer 6 mm diameter was used to drill holes 4 mm deep. Then 0.5 g of shampoo bar from each batches added in to this holes along with marketed products. Plates were then incubated at 37°C for 48 hr. The zone of inhibition around the well was measured and recorded.

Antifungal activity

Isolation of *Malassezia* species ^(18, 19)

Flakes or scales were collected from scalp by partitioning the hair with a sterile comb and scrapping approximately one inch area using a sterile blunt scalpel. The specimen was then transferred into a dark sampling paper to prevent exposure to sunlight. The samples were inoculated into of Sabouraud Dextrose broth tubes with gentamycin to avoid bacterial contaminants. The test tube was then incubated at 30 °C for 7 days, which were observed regularly.

The antifungal activity of all developed batches of formulation was carried out by Cup-plate method in comparison with marketed formulation. The fungal culture used was *Malassezia furfur*. The formulated samples were inoculated in the wells formed over the surface of Sabouraud Dextrose Agar (SDA) media. Plates were then incubated at 30°C for 5days. The zone of inhibition (diameter in mm) developed, if any, was then measured.

Stability study ⁽²⁰⁾

All the formulated shampoo bars were taken and kept for stability study at room temperature (30 ± 20°C) as well as refrigerator (4±20°C) over a period of one month.

4. Results

Physical evaluation was done by testing the colour, odour, appearance and texture of the shampoo bar. The shampoo bar was found to have pleasant odour and having red and green color. All the formulations were produced uniform distribution of extracts in shampoo bar. This was confirmed by visual appearance and by touch. Under visual inspection of the prepared formulation, it was indicated that no cracks and have uniform color dispersion. The results of foam height determination showed that the shampoo bar which is capable to produce high foaming property. ⁽²¹⁾ The determination of dirt dispersion indicated that the amount of ink in the foam was light in all six formulations, and no dirt

would stay in the foam. Dirt that stays in the foam will be difficult to rinse away and it will redeposit on the hair. Percentage solid content of all the formulations was ranges from 22.1% to 24.5%, which was in the acceptable range and hence they were easy to wash out from hair. The moisture content was used to estimate the percentage of water in the shampoo bar and all the formulated shampoo bar has ideal moisture content. All the shampoos were acid balanced and were ranged from 5.5 to 5.8, which is near to skin pH. Shampoos of lower pH are one of the ways to minimize damage of the hair. Mild acidity prevents swelling and promotes tightening of the scales, there by inducing shine^(22, 23)

Anti-bacterial activity

The antibacterial activity of the formulated shampoo bars was shown in table 2. The prepared anti-dandruff shampoo bars were subjected to antimicrobial activity using well-plate method in an agar medium. The zone of inhibition observed was shown that, all the formulation having anti-bacterial activity. From the results, the formulation F1 and F4 shows maximum activity compared to other formulations. In that, salicylic acid was showed more activity as compared to sulfur. The antimicrobial activity of the best formulations was compared with the marketed products and the results shows that shampoo bar containing salicylic acid and marketed product has more anti-bacterial activity as compared to sulfur (Table 3).

Table 2: Anti-bacterial activity study

Bacterial strain used	Salicylic acid	ZOI (mm)	Sulfur	ZOI (mm)
Bacillus subtilis	F1	5.6±0.2	F4	5.5±0.2
Bacillus subtilis	F2	5±0.1	F5	4.8±0.2
Bacillus subtilis	F3	4.8±0.1	F6	4.5±0.1

Table 3: Anti-bacterial activity in comparison with marketed product

S. no	Organism	Formulation	Zone of inhibition (mm)
1.	Bacillus subtilis	F1	5.5±0.3
2.	Bacillus subtilis	F4	5±0.2
3.	Bacillus subtilis	(Marketed product)	5.5±0.2

Anti-fungal activity

The anti-fungal activity of the prepared anti-dandruff shampoo bars were performed by using well-plate method in a sabouraud dextrose agar medium. The zone of inhibition observed was shown that, all the formulations having anti-fungal activity. From the results, the formulation F1 and F4 shows maximum activity compared to other formulations (Table 4). In that also salicylic acid containing formulation was showed more activity as compared to sulfur. The anti-dandruff activity of the prepared anti-dandruff shampoo bar formulations were compared with marketed product. The results indicated that shampoo bar containing salicylic acid and marketed product has more anti-dandruff activity as compared to sulfur (Table 5).

Table 4: Anti-fungal activity study

Fungal strain used	Salicylic acid	ZOI (mm)	Sulfur	ZOI (mm)
Malassezia	F1	5.5±0.2	F4	5.4±0.1
Malassezia	F2	4.9±0.2	F5	5±0.2
Malassezia	F3	5±0.3	F6	5±0.2

Table 5: Anti-dandruff activity in comparison with marketed product

Sl. no	Organism	formulation	Zone of inhibition (mm)
1.	Malassezia	F1 (Salicylic acid)	5.5±0.2
2.	Malassezia	F2 (sulfur)	4.9±0.1
3.	Malassezia	Marketed product	5.5±0.1

Stability study

The stability study was performed during a period of one month. After the study, all the formulations were subjected to evaluation. The results of pH, foam stability, moisture content was showed that the formulations were stable during the period. The anti-bacterial and antifungal activity were also performed and shown in table no 6 and 7. The results showed that there was no reduction in the activity⁽²⁴⁾

Table 6: Anti-bacterial activity after stability study

Organism	Salicylic acid	ZOI (mm)	Sulfur	ZOI (mm)
Bacillus subtilis	F1	5.5±0.2	F4	5.4±0.3
Bacillus subtilis	F2	5.2±0.2	F5	5.1±0.2
Bacillus subtilis	F3	5±0.2	F6	5±0.2

Table 7: Anti-fungal activity after stability study

Fungal strain used	Salicylic acid	ZOI (mm)	Sulfur	ZOI (mm)
Malassezia	F1	5.5±0.2	F4	5.4±0.1
Malassezia	F2	4.9±0.1	F5	5±0.2
Malassezia	F3	5±0.1	F6	5±0.1

5. Conclusion

In the present work, efforts have been made to prepare and evaluate anti-dandruff shampoo bar using two anti-dandruff agents such as salicylic acid, and sulfur by various combinations. Physical evaluation was done by testing the Colour, Odour, appearance and texture of the shampoo bar^(25, 26) The shampoo bar was found to have pleasant Odour and color. All formulations had the good characteristics with respect to foaming and acid balanced ranged 5.5 to 5.9, which is near to the skin pH. The result of solid content indicated that, they are easy to wash out and has moderate dirt dispersion property. Test for homogeneity shows that all formulations produce uniform distribution of extracts in shampoo bar. Under visual inspection of the prepared formulation indicated no cracks and have uniform color dispersion. The prepared anti-dandruff shampoo bar formulations F1 to F6 were subjected to antimicrobial activity (antibacterial and antifungal) using well-plate method. The zone of inhibition was observed successfully for formulation F1 and F4 than other formulations. The results show that shampoo bar containing salicylic acid has more activity as compared to sulfur.^(27, 28)

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