EVALUATION OF PREPARED SHAMPOO FORMULATIONS AND TO COMPARE FORMULATED SHAMPOO WITH MARKETED SHAMPOOS

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ABSTRACT

Shampooing is the most common form of hair treatment. Shampoos are primarily been products aimed at cleansing the hair and scalp. In the present scenario, it seems improbable that herbal shampoo, although better in performance and safer than the synthetic ones, will be popular with the consumers. A more radical approach in popularizing herbal shampoo would be to change the consumer expectations from a shampoo, with emphasis on safety and efficacy. We have evaluated and compared the herbal shampoo, which was formulated in previous study, with two marketed shampoos. The findings of this investigation reveal that synthetic preservatives have sometimes been the cause of adverse effects among consumers. We have used the physico-chemical approach to preservation and by formulating a self preserving shampoo, have avoided this risk posed by chemical preservatives. However, the aesthetic attributes, such as lather and clarity, of the laboratory shampoo are not comparable with the marketed shampoos. The foam volume was on a par. Although the retail products were not fare so well in the tests conducted by us, they enjoy market popularity, especially if they foam well. This is mainly due to the false notion among consumers that 'a shampoo that foams well, works well', and no real effort on the part of manufacturers to counter this fallacy.

Keywords: Herbal shampoo, radical approach, physico-chemical approach, aesthetic attributes, marketed shampoos.

INTRODUCTION

Evaluation of shampoos comprises the quality control tests including visual assessment and physiochemical controls such as pH, density and viscosity. Sodium lauryl sulfate based detergents are the most common but the concentration will vary considerably from brand to brand and even within a manufacturer's product range. Cheap shampoos may contain a high detergent concentration while expensive shampoos may contain very little of a cheap detergent[1].

Shampoos for oily hair can have exactly the same detergent at the same concentration as shampoos for dry hair. The difference is more likely to be a reduced amount of oil or conditioning agent in the shampoo for oily hair or the difference may even just be the packaging.

MATERIALS AND METHODS

Evaluation of Herbal Shampoos

To evaluate the prepared formulations, quality control tests including visual assessment and physicochemical controls such as pH, density and viscosity were performed. Also, to assure the quality of products, specific tests for shampoo formulations including the determination of dry residue and moisture content, total surfactant activity, salt content, surface tension, thermal and mechanical stability and detergency tests were carried out. The results were compared with marketed formulations[2].

1. **Physical appearance/visual inspection:** The formulations prepared were evaluated in terms of their clarity, foam producing ability and fluidity[2].
2. **Determination of pH:** The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C[3].
3. **Determine percent of solids contents:** A clean dry evaporating dish was weighed and added 4 grams of shampoo to the evaporating dish. The dish and shampoo was weighed. The exact weight of the shampoo was calculated only and put the evaporating dish with shampoo was placed on the hot plate until the liquid portion was evaporated. The weight of the shampoo only (solids) after drying was calculated.
4. **Rheological evaluations:** The viscosity of the shampoos was determined by using Brookfield Viscometer (Model DV-l Plus, LV, USA) set at different spindle speeds from 0.3 to 10 rpm[3]. The viscosity of the shampoos was measured by using spindle T_{95}. The temperature and sample container's size was kept constants during the study.
5. **Dirt dispersion:** Two drops of shampoo were added in a large test tube contain 10 ml of distilled water. 1 drop of India ink was added; the test tube was stoppered and shakes it ten times. The amount of ink in the foam was estimated as None, Light, Moderate, or Heavy.
6. **Cleaning action:** 5 grams of wool yarn were placed in grease, after that it was placed in 200 ml. of water containing 1 gram of shampoo in a flask. Temperature of water was maintained at 35^0C. The flask was shaked for 4 minutes at the rate of 50 times a minute. The solution was removed and sample was taken out, dried and weighed. The amount of grease removed was calculated by using the following equation:

$$DP = 100\left(1 - \frac{T}{C}\right)$$

In which, DP is the percentage of detergency power, C is the weight of sebum in the control sample and T is the weight of sebum in the test sample[4].

7. **Surface tension measurement:** Measurements were carried out with a 10% shampoo dilution in distilled water at room temperature. Thoroughly clean the stalagmometer using chronic acid and purified water. Because surface tension is highly affected with grease or other lubricants[5, 6]. The data calculated by following equation given bellow:

$$R_2 = \frac{(W_3 - W_1)\, n_1}{(W_2 - W_1)\, n_2} \times R_1$$

where W_1 is weight of empty beaker.

W_2 is weight of beaker with distilled water.

W_3 is Weight of beaker with shampoo solution.

n_1 is no. of drops of distilled water.

n_2 is no. of drops of shampoo solution.

R_1 is surface tension of distilled water at room temperature.

R_2 is surface tension of shampoo solution.

8. **Detergency ability:** The Thompson method was used to evaluate the detergency ability of the samples. Briefly, a crumple of hair were washed with a 5% sodium lauryl sulfate (SLS) solution, then dried and divided into 3g weight groups. The samples were suspended in a n-hexane solution containing 10% artificial sebum and the mixture was shaken for 15 minutes at room temperature. Then samples were removed, the solvent was evaporated at room temperature and their sebum content determined. In the next step, each sample was divided into two equal parts, one washed with 0.1 ml of the 10% test shampoo and the other considered as the negative control. After drying, the resided sebum on samples was extracted with 20 ml n-hexane and re-weighed. Finally, the percentage of detergency power was calculated using the following equation:

$$DP = 100\left(1 - \frac{T}{C}\right)$$

In which, DP is the percentage of detergency power, C is the weight of sebum in the control sample and T is the weight of sebum in the test sample[3, 4].

9. **Foaming ability and foam stability:** Cylinder shake method was used for determining foaming ability. 50 ml of the 1% shampoo solution was put into a 250 ml graduated cylinder and covered the cylinder with hand and shaken for 10 times. The total volumes of the foam contents after 1 minute shaking were recorded. The foam volume was calculated only. Immediately after shaking the volume of foam at 1 minute intervals for 4 minutes were recorded[7].

10. **Skin sensitization test:** The guinea pigs were divided into 7 groups (n=3). On the previous day of the experiment, the hairs on the backside area of guinea pigs were removed. The animals of group I was served as normal, without any treatment. Animal Group II, III, IV, V and VI were applied with shampoo formulation F1, F2, F3, MS1 and MS2 respectively. Shampoos were applied onto nude skin of animals of groups. A 0.8% v/v aqueous solution of formalin was applied as a standard irritant on animal Group VII. The animals were applied with new patch/formalin solution up to 72 hours and finally the application sites were graded according to a visual scoring scale, always by the same investigator. The erythema scale was as follows: 0, none; 1, slight; 2, well defined; 3, moderate; and 4, scar formation (severe)[4].

11. **Eye irritation test:** Animals (albino rats) were collected from animal house. About 1% shampoo solutions was dripped into the eyes of six albino rabbits with their eyes held open with clips at the lid. The progressive damage to the rabbit's eyes was recorded at specific intervals over an average period of 4 seconds. Reactions to the irritants can include swelling of the eyelid, inflammation of the iris, ulceration, hemorrhaging (bleeding) and blindness[4].

12. **Surface characterization:** Surface morphology of the hairs was examined by scanning electron microscopy (Leo 430, Leo Electron Microscopy Ltd., Cambridge, England). The hair samples were mounted directly on the SEM sample stub, using double side stitching tape and coated with gold film (thickness 200nm) under reduced pressure (0.001 mm of Hg). The photomicrographs of suitable magnification were obtained for surface characterization. The following seven samples were characterized by SEM:

Sample-1: Hairs

Sample-2: Hairs with sebum

Sample-3: Hairs with sebum and washed with shampoo*, F_1

Sample-4: Hairs with sebum and washed with shampoo*, F_2

Sample-5: Hairs with sebum and washed with shampoo*, F_3

Sample-6: Hairs with sebum and washed with shampoo, MS_1

Sample-7: Hairs with sebum and washed with shampoo, MS_2

(*shampoo means 10% w/v formulation in distilled water)

13. **Stability studies:** The thermal stability of formulations was studied by placing in glass tubes and they were placed in a humidity chamber at 45°C and 75% relative humidity. Their appearance and physical stability were inspected for a period of 3 months at interval of one month[8, 9].

Table 1: Evaluation of Formulation for physical appearance, pH and Solids

S. No.	Formulation	Physical Appearance	pH	Solids (%)
1	F1	Dark brown, good foaming	5.51± 0.02	22.11± 0.02
2	F2	Dark brown, good foaming	5.53± 0.07	24.51± 0.02
3	F3	Dark brown, good foaming	5.61± 0.02	29.31± 0.02
4	MS1	Light brown, good foaming	5.81± 0.04	25.41± 0.02
5	MS2	Light green, good foaming	5.91± 0.01	28.21± 0.02

Table 2: Viscosities of herbal shampoos

Speed (rpm)	F1		F2		F3		MS1		MS2	
	%Tor	Viscosity	%Tor	Viscosity	%Tor	Viscosity	%Tor	Viscosity	%Tor	Viscosity
0.3	15.31	95733.33	-	-	13.35	83433.33	-	-	-	-
0.5	21.90	82150.00	16.18	60765.00	19.61	73583.33	-	-	-	-
1	32.86	54150.00	22.75	42666.66	27.46	51516.67	-	-	10.26	17533.33
1.5	40.73	50916.67	26.66	33350.00	32.35	40450.00	-	-	11.93	14916.67
2.5	51.70	38778.33	31.96	23978.33	38.46	28851.66	10.13	7598.33	15.30	11478.33
5	67.63	25425.00	38.98	14645.33	47.03	17651.66	15.85	5945.00	24.41	9158.33
10	84.53	15775.00	46.40	8541.00	57.28	10741.66	27.50	5156.67	41.41	7766.66

Table 3: Evaluation of Formulation for cleaning, Surface tension and Detergency

S. No.	Formulation	Cleaning (%)	Surface tension (dynes/cm)	Detergency (%)
1	F1	24.21± 0.03	32.15± 0.02	64.23± 0.32
2	F2	32.51± 0.09	33.22± 0.12	65.12± 0.02
3	F3	18.81± 0.08	31.37± 0.62	53.58± 0.09
4	MS1	33.61± 0.05	34.60± 0.32	67.69± 0.12
5	MS2	32.11± 0.02	33.61± 0.42	66.12± 0.42

Table 4: Foam stability of herbal shampoos

Time (minutes)	Foam volume (ml)				
	F1	F2	F3	MS_1	MS_1
1	170	180	140	180	180
2	168	177	137	178	177
3	166	175	134	176	174
4	165	174	135	175	173
5	164	173	134	174	172

Table 5: Stability studies herbal formulations

Parameters	1month	2month	3month
Physical appearance/visual inspection	Clear	Clear	Clear
pH	5.51± 0.02	5.53± 1.02	5.61± 0.82
Solids contents (%)	22.51± 0.02	24.11± 0.92	26.51± 1.02
Surface tension measurement (dy. /cm)	33.22± 0.12	32.52± 0.32	35.20± 0.72
Rheological evaluations (cps)	94607.89	30647.63	57749.44
Detergency ability (%)	65.12± 0.12	67.10± 0.10	54.11± 0.52
Foaming ability and foam stability (ml)	170	180	170

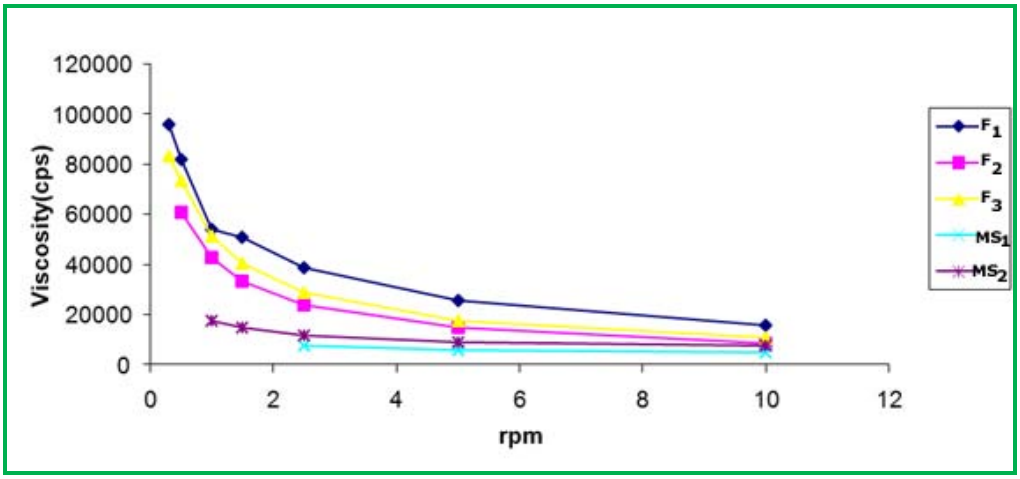

Figure 1: Viscosity profile of herbal shampoos

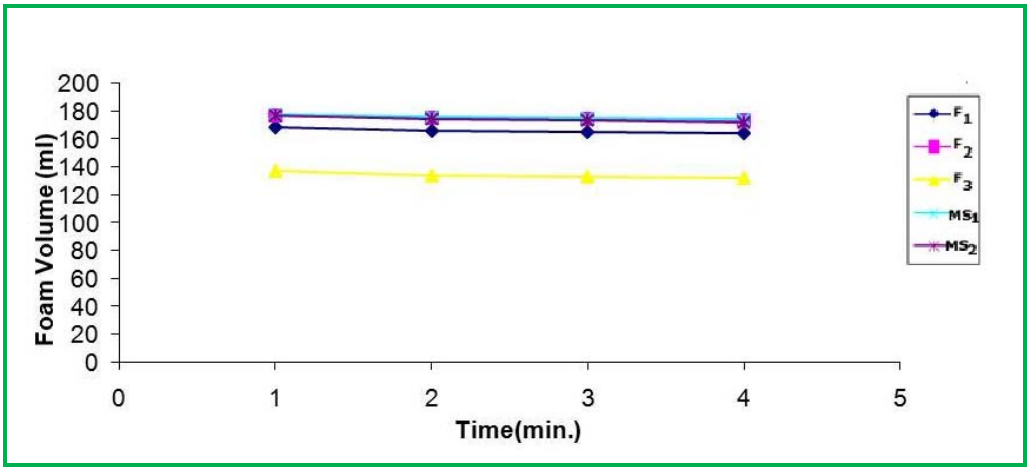

Figure 2: Foam retention profiles of herbal shampoos

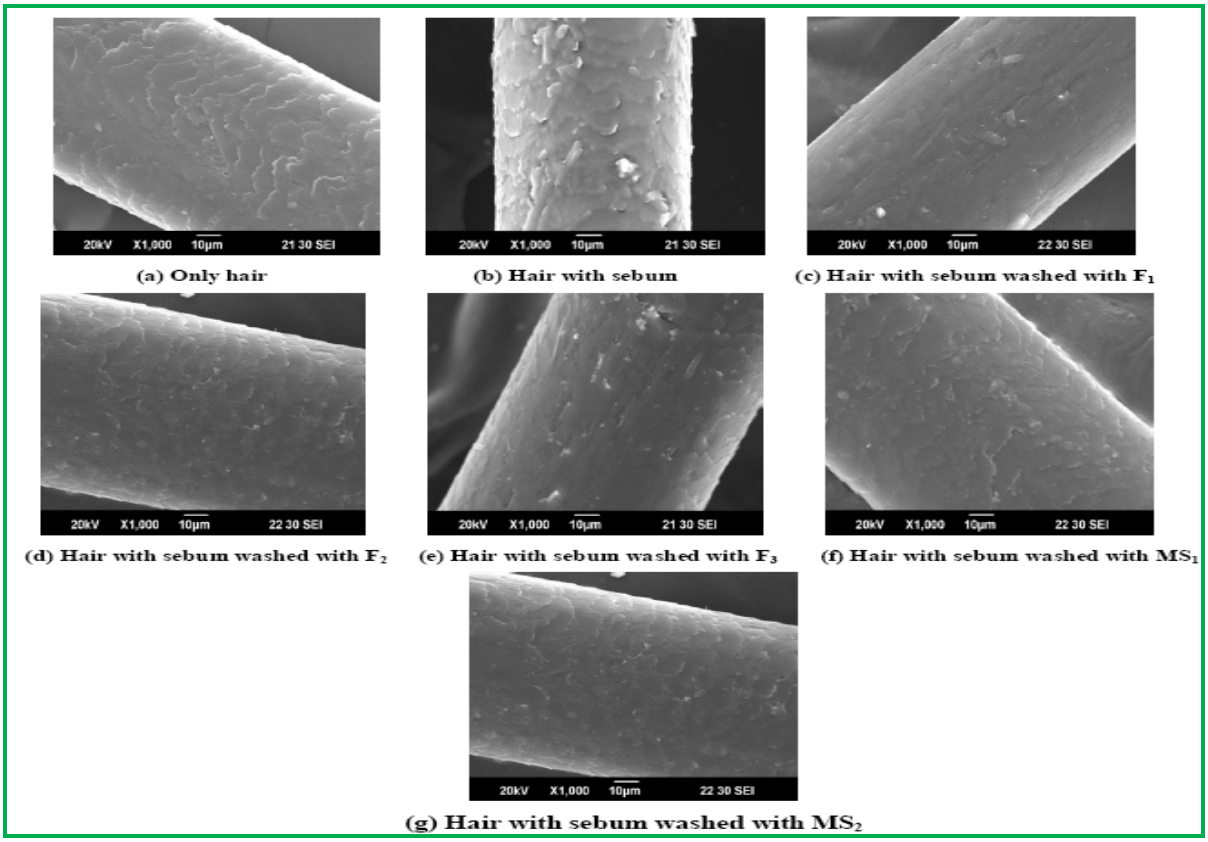

Figure 3: SEM Photograph of Formulations

RESULTS AND DISCUSSION

Evaluation of Herbal Shampoos

1. Physical Appearance/Visual Inspection

The results of visual inspection of series of formulations are listed in Table 1. As can be seen, all formulations had the good characteristics with respect to foaming.

2. pH

The pH of shampoos has been shown to be important for improving and enhancing the qualities of hair, minimizing irritation to the eyes and stabilizing the ecological balance of the scalp[10]. The current trend to promote shampoos of lower pH is one of the ways to minimize damage to the hair. Mild acidity prevents swelling and promotes tightening of the scales, there by inducing shine. As seen from Table 1, all the shampoos were acid balanced and were ranged 5.5 to 5.9, which is near to the skin pH.

3. Percent of Solids Contents

If the shampoo has too many solids it will be hard to work into the hair or too hard to wash out. The result of percent of solids contents is tabulated in table 1, and was found between 22-29%. As a result, they were easy to wash out.

4. Rheological evaluations

The results of rheological evaluation showed that the viscosity of the samples changes gradually with the increase in rpm, therefore the shampoo formulations were time dependent. Secondly as the data showed the viscosity decreases with increase in rpm, so the shampoo formulations were shear thinning or pseudo plastic in nature. These formulations showed pseudo plastic behavior which is a desirable attribute in shampoos formulation. At low rpm the herbal shampoos showed high viscosity and increase in the shear rate the viscosity of the shampoos drops, this is a favorable property which eases the spreading of the shampoos on hair. The results obtained from the rheological studies were fitted into different flow behaviors, using the linear or non-linear regression. Table 2 shows the goodness of fitting indices for Newtonian, plastic and pseudo plastic flow behaviors. As can be seen in the Table 2, all the formulations followed a pseudo plastic rheogram. As the Figure 1 shows that the graphical lines are not linear line so the formulations are Non-Newtonian in nature.

5. Dirt Dispersion

Shampoo that cause the ink to concentrate in the foam is considered poor quality, the dirt should stay in water. Dirt that stays in the foam will be difficult to rinse away. It will redeposit on the hair. All five shampoos showed similar results. These results indicate that no dirt would stays in the foam; so prepared and marketed formulations are satisfactory.

6. Cleaning Action

Cleaning action was tested on wool yarn in grease. Although cleaning or soil/sebum removal is the primary aim of a shampoo, experimental detergency evaluation has been difficult to standardize, as there is no real agreement on a standard soil, a reproducible soiling process or the amount of soil a shampoo should ideally remove[11]. As seen from the results, there is a significant difference in the amount of sebum removed by the different shampoos. The results of detergency studies showed that the final formulation has significantly similar detergency ability, when compared with the marketed formulations and it was found in between 18-33%. The results are presented in Table 3.

7. Surface tension measurement

It has been mentioned that a proper shampoo should be able to decrease the surface tension of pure water to about 40 dynes/cm[12]. Surface tension reduction is one of the mechanisms implicated in detergency. The reduction in surface tension of water from 72.8 dynes/cm to 35.37 dynes/ cm by the herbal shampoos is an indication of their good detergent action. The results are shown in Table 3.

8. Detergency ability

Although cleaning or soil/sebum removal is the primary aim of a shampoo, experimental detergency evaluation has been difficult to standardize, as there is no real agreement on a standard soil, a reproducible soiling process or the amount of soil a shampoo should ideally remove. As seen from the results, there is a significant difference in the amount of sebum removed by the different shampoos. Shampoo MS_1, MS_2 being a frequent-use cleanser, was expected to have the maximum detergency. Shampoos F_1, F_2 and F_3 also showed moderate detergency. The results are presented in Table 3.

9. Foaming ability and foam stability

Although foam generation has little to do with the cleansing ability of shampoos, it is of paramount importance to the consumer and is therefore an important criterion in evaluating shampoos. All the five shampoos showed similar foaming characteristics in distilled water. The foam retention ability of five samples is given in figure 2. All five shampoos showed comparable foaming properties. The foam stability of herbal shampoos is listed in table 4. A point to be noted here is that there does not seem to be any direct correlation between detergency and foaming, which only confirms the fact that a shampoo that foams well need not clean well. The final formulation produced stable foams there was little bet change in foam volume.

10. Skin Sensitization Test

In case of cosmetics containing higher percentage of potential irritants like hair dyes, shampoos, hair tonics and patches should not be sealed. These should be used as open patches. There were no hypersensitive reactions by those formulations. All formulations are good.

11. Eye Irritation Test

The all formulation showed no eye irritation after 2 seconds but light irritation showed after 4 second of treatment by all formulation including marketed shampoos.. The adverse reactions may occur to one of the primary constituents of the cosmetic formulation or contamination or procedural misconduct. Preservatives are the second most common cause of skin reactions besides

fragrances. In most cases, these are only mild or transient such as stinging and smarting and contact urticarial. In few cases, reactions may be more severe with redness, edema, dryness and scaling. There were no eyes irritations by all formulations. All formulations were good.

12. Surface Characterization by SEM

Digital pictures were obtained by SEM, at a magnification of 1000x. Hair treated with shampoo the scales were regularly oriented and characterized by homogeneous borders. There was no groove along the hair long axis. The Figure 3 obtained by SEM showed clear differences between treated and untreated hairs. The SEM was successfully used to study the effects of treatment with shampoos on the microstructure of hair. This investigates described the use of SEM for comparing the cleansing and condensing effects of herbal shampoos. It's important to understand the cleansing action of shampoos by removal of deposited artificial sebum on hairs[12]. In addition to detergency evaluation surface morphology studies were performed to correlate the results obtain from detergency removal test. The SEM photographs of Figure 3 compared untreated hair and hair treated with formulated and marketed shampoos. The untreated hair had a scaled appearance due to desquamation of the hair cuticle. SEM observations provided the evidence for cleansing action and were in accordance to the results obtained from detergency and cleansing action. Formulated as well as marketed shampoos possessed good cleansing property as reviewed by micro photographs (Figure 3). The formulated shampoos were found to have a good overall cleansing action, however this action lesser was then marketed shampoos. Hence the treatment of hairs with shampoos containing natural conditioners (Aloe vera, Orange) protects hair morphological integrity, provided consistency and shin to the hair and easy combing. The cleansing action in the herbal shampoos was provided by active natural foam forming agents that is saponins. These shampoos also contain phenolic substances along with vitamin-C that provides anti-oxidant effects. As a result of free-radical scavenging effect and lower potential alteration in hair morphology was prevented. Thus hairs become protected against the cognitive action of brushing. It can be concluded from SEM studies that natural plants extract present in formulated and marketed shampoos play an important role in protecting hair structures along with cleansing and conditioning effects.

13. Stability Study

Stability and acceptability of organoleptic properties (odor and color) of formulations during the storage period indicated that they are chemically and physically stable. The stability of herbal formulation is listed in table 5.

CONCLUSION

The formulated shampoos were not only safer than the chemical conditioning agents, but also greatly reduce the protein loss during combing. The pH of the shampoos was adjusted to 5.5, to retain the acidic mantle of scalp. Synthetic preservatives have sometimes been the cause of adverse effects among consumers. We have used the physico-chemical approach to preservation and by formulating a self preserving shampoo, have avoided this risk posed by chemical preservatives. However, the aesthetic attributes, such as lather and clarity, of the laboratory shampoo are not comparable with the marketed shampoos. The foam volume is on a par. Although the retail products do not fare so well in the tests conducted by us, they enjoy market popularity, especially if they foam well. This is mainly due to the false notion among consumers that 'a shampoo that foams well, works well', and no real effort on the part of manufacturers to counter this fallacy.

In the present scenario, it seems improbable that herbal shampoo, although better in performance and safer than the synthetic ones, will be popular with the consumers. A more radical approach in popularizing herbal shampoo would be to change the consumer expectations from a shampoo, with emphasis on safety and efficacy. Formulators must play an active role in educating the consumers about the potential harmful effects of synthetic detergents and other chemical additives present in shampoos. There is a strong need to change the consumer perception of a good shampoo and the onus lies with the formulators.

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