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FORMULATION AND EVALUATION OF POLY HERBAL SHAMPOO

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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 marketed shampoo. The findings of this investigation reveal that synthetic preservatives have sometimes been the cause of adverse effects among consumers. 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
Shampoo is defined as a preparation of a surfactant (surface active material) in suitable form liquid solid or power which when used under the conditions specified will remove surface grease, dirt an skin debris from the hair shaft and scalp without affecting adversely the hair, scalp or health of the user. Shampoo is a hair care product used for the removal of oils, dirt, skin particles, dandruff, environmental pollutants and other contaminant particles that gradually build up in hair. The goal is to remove the unwanted build-up without stripping out so much sebum as to make hair unmanageable. 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. 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. The added functions of shampoo include lubrication, conditioning, and medication. A shampoo is used as a cosmetic preparation meant for the washing of hair and scalp, cleansing the hair of accumulated sebum.[1,2]

MATERIALS AND METHODS

S. No	Materials	Company name
1	Neem oil	Shree Baidyanath Ayurvedic Bhawan , Mumbai
2	Amla oil	Hamdard Laboratories, Mumbai
3	Alovera gel	Cavin Care, Chennai
4	Mandaram oil	Sri Chintamani Pharmaceutical works,Tenali
5	Sodiumlara sulphate	S.D Fine Chemicals, Mumbai
6	Glycerin	S.D Fine Chemicals, Mumbai
7	EDTA	S.D Fine Chemicals, Mumbai
8	Sodium Hydroxide	S.D Fine Chemicals, Mumbai

METHODS

Formulation of poly herbal shampoo[3]

The Poly herbal shampoo was formulated using simple mixing process. Herbal anti- drandruff shampoo was formulated by adding the required amounts of herbal ingredients as given in the formulation table 1.

Table 1 Formulation of Shampoo.

Ingredients	F1	F2	F3	F4	F5
Neem oil	1ml	1ml	0.5ml	0.5ml	0.5ml
Amla oil	1ml	1ml	0.5ml	0.5ml	0.5ml
Alovera gel	10mg	10mg	10mg	10mg	10mg
Mandaram oil	1ml	1ml	0.5ml	0.5ml	0.5ml
Sodium lauryl sulphate	10mg	12mg	15mg	18mg	20mg
Glycerin	1ml	1ml	1ml	1ml	1ml
EDTA	0.15mg	0.15mg	0.15mg	0.15mg	0.15mg
Sodium hydroxide	To adjust P^H	To adjust P^H	To adjust P^H	To adjust P^H	To adjust P^H
Perfume	0.1ml	01ml.	0.1ml	0.1ml	0.1ml

Evaluation of Poly Herbal Shampoo[4]

Physical Appearance/Visual Inspection[5]

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

Determination of percent of solids contents[6]

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.

Dirt dispersion[7,8]

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.

Surface Tension Measurement[9,10]

Measurements were carried out with a 10% shampoo dilution in distilled water at room temperature.The stalagmometer will be cleaned using chromic acid and purified water because surface tension it will be highly affected with grease or other lubricants. The data was calculated by following equation given below.

$$R2 = \frac{(W3-W1)\, n1}{(W2-W1)\, n2} \times R1$$

where W1 is weight of empty beaker.
W2 is weight of beaker with distilled water.
W3 is Weight of beaker with shampoo solution.
n1 is no. of drops of distilled water.
n2 is no. of drops of shampoo solution.
R1 is surface tension of distilled water at room temperature.
R2 is surface tension of shampoo solution.

Cleaning Action

5 grams of hair were placed in soil, after that it was placed in 200 ml. of water containing 1 gram of shampoo in a flask. Temperature of water was maintained at 350C. 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.

$$DP = 100\,(1 - T / C)$$

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 sample4.

Measurement of P^H[11]

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

Wetting Time[12,13]

The canvas was cut into 1 inch diameter discs having an average weight of 0.44 gms. The disc was floated on the surface of the shampoo solution 1% w/v and the stopwatch started. The time required for the disc to begin to sink was measured accurately and noted as the wetting time.

Foaming Ability and Foam Stability[14]

Cylinder shake method was used for determining foaming ability. 50 ml of the 1% shampoo solution was put into a 250ml 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.

Measurement of Viscosity[15]

The viscosity profile of the shampoo formulation was measured using Brookfield viscometer DV-E model at 25° C. (Brookfield engineering labs, USA)

Skin Sensitization Test[16]

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 wasserved as normal, without any treatment. Animal Group II, III, IV, V and VI were applied with shampoo formulation F1, F2, F3, F4 and F5 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 formalin solution up to 8 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).

Eye Irritation Test[17]

Animals (albino rabbits) 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 blindness4.

Antifungal activity[18]

By direct pouring method

Sample preparation: Each shampoo in the study was used in its concentrated form and in 10-fold serial dilutions using sterile distilled water up to a dilution of 103. Sabouraud"s dextrose agar (1.5% agar) with 1% olive oil added to the medium was used throughout the study. The oil was mixed prior to autoclaving at 15 psi for 15 min. The autoclaved medium was allowed to cool and then sufficient medium was transfer into Petri dishes. Two ml of the each shampoo dilutions and one ml of the each organism suspension were also added into the Petri dishes and gently rocked to disperse all three ingredients. All plates were incubated at 37 0 C. After five days incubation at 37^0 C, all plates were read for growth.[13]

Stability Studies

Stability studies were carried out by placing glass tubes in humidity chamber at 45°C and 75% relative humidity. And their appearance, physical stability were inspected for a period of 3 months at interval of one month.

RESULTS AND DISCUSSION

Physical appearance/visual inspection

The results of visual inspection of series of formulations are listed in Table 2. As can be seen, all formulations had the good characteristics with respect to foaming and colorful appearance.

Table 2: Evaluation of formulations for Physical Appearance.

S.No	Formulation	Physical Appearance
1.	F1	Off Green,good Foaming
2.	F2	Off Green,good Foaming
3.	F3	Off Green,good Foaming
4.	F4	Off Green,good Foaming
5.	F5	Off Green,good Foaming
6.	F0 (Marketed Formulation)	Off Green, good Foaming

Fig 1: Prepared 5poly herbal formulations.

Determination of 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 3, and was found between 21-29%. As a result, they were easy to wash out.

Table 3: Evaluation of formulations for % Solids.

S.No	Formulation	% Solids
1	F1	21.38±0.02
2	F2	24.02±0.03
3	F3	26.25±0.02
4	F4	27.58±0.04
5	F5	29.03±0.02
6	F0 (Marketed Formulation)	28.32±0.03

Fig 2: Determining % Solid Content using Hot Plate.

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 six shampoos (F1-F5) and F0 showed similar results as shown in stable 4. These results indicate that no dirt would stays in the foam; so prepared and marketed formulations are satisfactory.

Table 4: Evaluation of formulations for Dirt Dispersion

S.No	Formulation	Dirt dispersion
1	F1	Moderate
2	F2	Light
3	F3	Light
4	F4	Light
5	F5	Not Found
6	F0 (Marketed Formulation)	Not Found

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. Surface tension reduction is one of the mechanisms implicated in detergency. The reduction in surface tension of water from 72.8 dynes/cm to 37.48 dynes/ cm by the herbal shampoos is an indication of their good detergent action. The results are shown in Table 5.

Table 5: Evaluation of formulations for Surface tension.

S.No	Formulation	Surface Tension (dynes/cm)
1.	F1	31.10±0.62
2.	F2	31.98±0.02
3.	F3	32.45±0.12
4.	F4	32.37±0.34
5.	F5	31.87±0.26
6.	F0 (Marketed Formulation)	33.16±0.42

Cleaning Action[23]

Cleaning action was tested on hair with soil. 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 as shown I the figure 8 and 9. 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 23-34%. The results are presented in Table 6.

Table 6: Evaluation of formulations for Cleaning action.

S.No	Formulation	Cleaning %
1.	F1	23.15±0.09
2.	F2	32.83±0.18
3.	F3	32.76±0.17
4.	F4	33.32±0.03
5.	F5	34.56±0.06
6.	F0 (Marketed Formulation)	34.78±0.07

Fig 3&4 Showing the Cleaning action of shampoos.

Measurement of P^H

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. 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 7, all the shampoos were acid balanced and were ranged 5.2 to 5.9, which is near to the skin pH.

Table 7: Evaluation of formulations for P^H:

S.No	Formulation	P^H
1.	F1	5.21±0.02
2.	F2	5.49±0.02
3.	F3	5.51±0.01
4.	F4	5.72±0.07
5.	F5	5.83±0.02
6.	F0 (Marketed Formulation)	5.91±0.004

Wetting Time

The wetting ability of a substance is a function of its concentration. By comparing the results from the table 8 it seems to be all the formulations are having optimum wetting capacity. Among all the formulations F5 is showing less wetting time. But compared to the marketed formulation among all the five formulations F2, F4 and F5 showed less wetting time showing that F0 is having moderate wetting capacity.

Table 8: Evaluation of formulations for Wetting Time.

S.No	Formulation	Wetting time (Sec)
1.	F1	224±0.32
2.	F2	178±0.45
3.	F3	183±0.22
4.	F4	171±0.56
5.	F5	158±0.36
6.	F0 (Marketed Formulation)	179±0.20

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 Six shampoos showed similar foaming characteristics in distilled water. The foam retention ability of five samples is given in table 9. All Six shampoos showed comparable foaming properties. 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.

Table 9: Evaluation of formulations for Foaming Ability and Foam Stability

TIME	F1	F2	F3	F4	F5	F0 (MF)
1 min	212	221	223	225	230	228
2 min	209	219	221	225	226	225
3 min	206	218	220	221	223	222
4 min	204	217	219	220	220	221
5 min	203	216	218	219	219	218

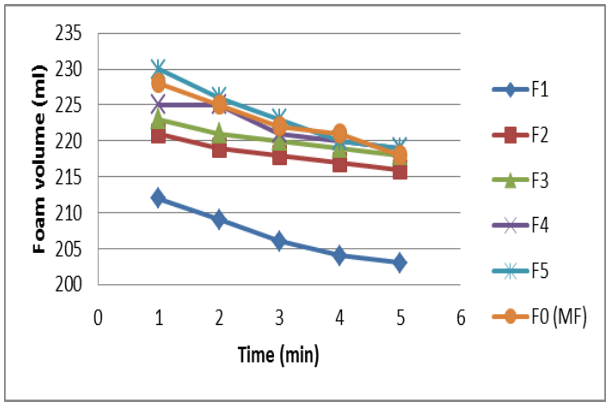

Fig 5: Foam retention profiles of herbal shampoos.

Measurement of Viscosity

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 10 shows the goodness of fitting indices for Newtonian, plastic and pseudo plastic flow behaviors. As can be seen in the Table 10, all the formulations followed a pseudo plastic rheogram.

Table 9: Evaluation of formulations for Foaming Ability and Foam Stability

RPM	F1	F2	F3	F4	F5	F0 (MF)
0.5	73793.00	76854.56	69896.15	65894.85	61356.33	--
1.0	60576.63	71256.32	65893.54	47895.32	45879.21	--
2.5	50879.79	53245.69	47812.32	36215.89	31256.89	17483.23
5	28796.23	21254.56	21569.02	18564.36	14568.91	13215.56
10	15876.25	11236.32	10256.32	11023.54	12551.23	7145.36

Skin Sensitization Test

The all formulations showed no skin sensitization reaction including marketed formulation. There were no hypersensitive reactions by those formulations. All formulations are good.

Fig 6.Initial Skin condition of Rabbit

Fig 7. Skin condition of Rabbit after 1 hr.

Fig 8: Skin condition of Rabbit after 3 hr

Eye Irritation Test

There were no eyes irritations, dryness or scaling by all formulations. All formulations were good. 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.

Fig 9: Initial eye condition of rabbit without applying shampoo.

Fig 10: Eye irritation test result after 30 seconds for Formulation F1.

Fig 11: Eye irritation test result after 30 seconds for Formulation F2.

Fig 12: Eye irritation test result after 30 seconds for Formulation F3.

Fig 13: Eye irritation test result after 30 seconds for Formulation F4.

Fig 14: Eye irritation test result after 30 seconds for Formulation F4.

Table 10: Evaluation of Formulations for Ski Irritation test.

Formulation	Skin irritation and eye irritation
F1	NO *Irritation*
F2	NO *Irritation*
F3	NO *Irritation*
F4	NO *Irritation*
F5	NO *Irritation*

Antifungal Activity

Among all the formulations F2, F4 and F5 were subjected to antifungal activity. Among all the formulations F2 and F5 showed maximum zone of inhibition. Therefore it is concluded that though the concentration of the herbs in all the formulations is same depending upon the formulation specifications with other ingredients the potential activity was varied between the formulations. Anti fungal activity for all the above three samples was found to be significant from this preliminary study. But herbal extracts activity is less active than standard Fluconazole. All the values are tabulated under Table 11.

Table 11.Evaluation of formulations for Antifungal Activity.

Sample code	Zone of Inhibition (in mm) average of 3	Remarks
F2	29	Significant
F4	25	Significant
F5	28	Significant

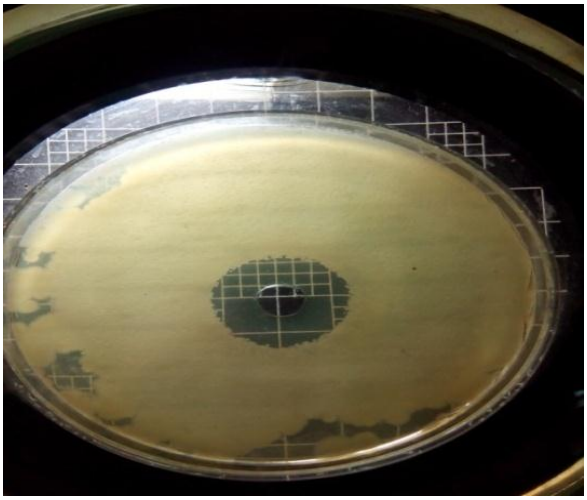

Fig 16. Antifungal Activity of Formulation F2.

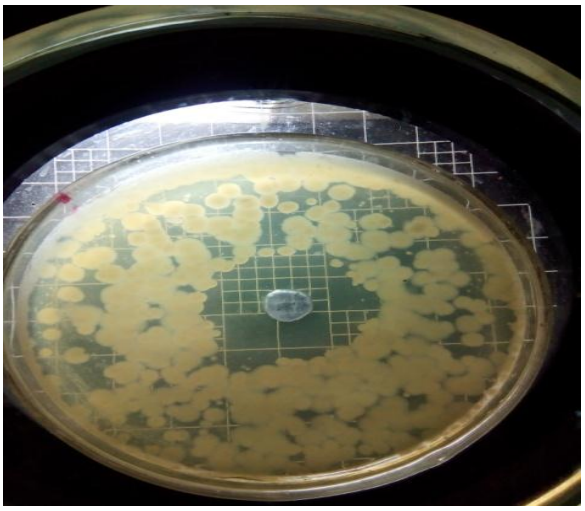

Fig 17: Antifungal Activity of Formulation F4.

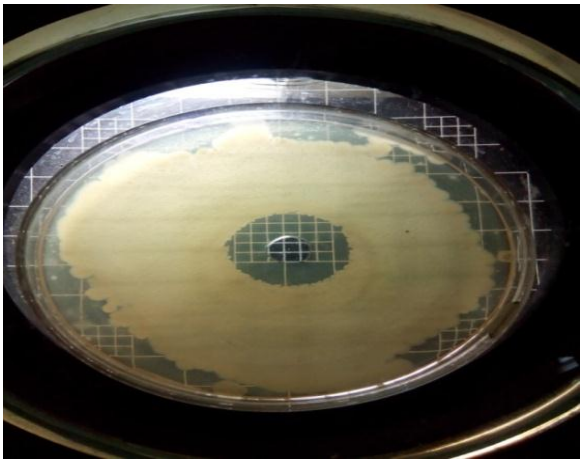

Fig 18: Antifungal Activity of Formulation F5.

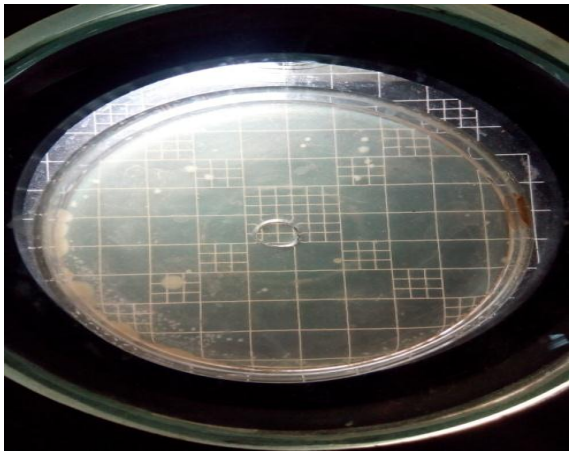

Fig 19: Antifungal Activity of –Ve Control.

Stability Studies

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 12.

Table 12: Stability studies herbal formulations.

Parmeters	1 month	2 month	3 month
Physical appearance/visual inspection	Clear	Clear	Clear
pH	5.83±0.02	5.83±0.58	5.83±0.83
Solid Contents (%)	29.03±0.02	29.03±0.02	29.81±0.01
Surface tension (dynes /cm)	30.83±0.04	30.56±0.05	30.56±0.02
Viscocity (cp)	12551.32	12487.25	11986.78
Dirt Dispersion	None	None	None
Foaming Volume	218	216	215

SUMMARY AND CONCLUSION

From the view of above experimental work where 5 formulations were prepared, the formulations showed good consistency, foaming. The visual inspection of the shampoos prepared were satisfactory. All the formulations had the good characteristics with respect to foaming. The P^H of shampoos has been shown to be important for improving and enhancing the qualities of hair, minimizing the irritation of eyes and stabilizing the ecological balance of the scalp. As seen from the P^H values resulted all the shampoos were acid balanced and were ranged 5.1-5.8, which is near to skin P^H .If the shampoo has too many solids it will be hard to work into the hair or too hard to wash out. All the formulations have given satisfactory results for the % solid contents. As aresult they were easy to wash out.It has mentioned proper shampoo should able to decrease the surface tension of pure water to about 40 dynes/cm.

The reduction in surface tension of water from 72.8 dynes /cm to 34.8 dynes /cm by the herbal shampoos is an indication of their good detergent action. Cleaning action was tested on hair sample taken along with soil. As seen from the results, there is a significant difference in the amount of soil removed by the different shampoos. The foam stability of herbal shampoos is resulted. The results of antifungal activity are described in table no 11 and zone of inhibition was clearly showed in fig 16-19..The formulation F2 and F5 shown the best zone of inhibition.. It is concluded that no change in the concentration of ingredients have not shown much popular difference in the antifungal activity. All the evaluation parameters were compared with marketed formulation (Garnier Frutis). Eye irritation was absent for all the formulations. From all the evaluated results the all the formulations had shown soncisted results with no much difference between them and also with marketed formulation. But compared to all F5 had shown better stating results with no dirt, good foaming stability and optimum anti fungal activity. So F5 was subjected to preliminary stability studies and evaluated for 3 months for its stability which showed lesser changes in its stability.

The formulated shampoos were not only safer than the chemical conditioning agents, but also greatly reduce the protein loss during combing. 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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