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FORMULATION AND EVALUATION OF POLYHERBAL LIQUID SHAMPOO

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ABSTRACT
A shampoo may be described as a cosmetic preparation meant for the washing of hair and scalp. Its primary function is of cleansing the hair of accumulated sebum, scalp debris and residue of hair grooming preparation. In the present scenario, the herbal shampoo is better in performance and safer than the synthetic ones. The herbal shampoo was formulated by adding Acacia *concinna*, *Sapindus mukorossi*, Aloe *barbadensia*, *Trigonella foencum*, *Phyllanthus emblica*, *Azadirachta indica*, *Hibiscus rosea sinesis* and *Lawsonia inermis*. The combination of such ingredients of herbal origin had made it possible to secure high effective shampoo. Different shampoo formulation at laboratory scale was done easily and evaluated for number of parameters to ensure its safety and efficacy.

KEYWORDS: Herbal shampoo, viscosity, Detergency Power.

INTRODUCTION
Today a large population both man and women use shampoo for washing the hair. One of the main functions of shampoo is to remove dirt on the hair. The dirt consist of sebum by the scalp, sweat residue, flakes of horny layer and residue from hair care cosmetics, dust and other external matter settled on the hair. (Ross et al., 1941)Various synthetic shampoo derivatives have been proved to cause harmful effect. Nowadays people are having awareness of their effects on hair. (Ross et al., 1941) Due to these reasons the community is getting attracted towards herbal products due to their inexpensive nature and negligible side effects. Nowadays, the usefulness of herbs in the cosmeceutical production has been extensively increased and there is a great demand for the herbal cosmetics. This polyherbal shampoo was formulated using nature ingredients like *Acacia concinna, Sapindus mukorossi, Aloe barbadensia, Trigonella foencum, Phyllanthus emblica, Azadirachta indica, Hibiscus rosea sinesis* and *Lawsonia inermis*. (Nafisy et al., 1989)

MATERIALS AND METHODS
Different parts of plants were selected to study hair care property.

Collection of Plants
Fresh parts of Hibiscus rosea sinesis (hibiscus leaves and flowers), *Azadirachta indica* (neem leaves), *Lawsonia inermis* (heena leaves) and aloe leaves were collected from botanical garden and washed under running water to remove contaminants while *Sapindus mukorossi* (reetha), *Acacia concinna* (shikkaai), *Phyllanthus emblica* (amla) and *Trigonella foencum* (methi seeds) were collected in dried form from market and then authenticated from department of botany, Vinodini PG college, Shekhawati university, Rajasthan. All the green fresh ingredients were dried in shade, converted into coarse powders and sieved. The raw materials were given with their biological source and uses respectively in table no. 1.

Methods of Preparation
Decoction of all herbal constituents in one part of water while Sodium Lauryl Sulphate was mixed with other part of water.

Procedure
Fenugreek seeds, reetha, shikkaai and amla were placed in a stainless steel and poured distilled water and kept aside for 24 hours. After that the mixture kept on boiling with excess of water and all other herbal constituents until the water reduced to one quarter then strained and filtered. Filtered product is concentrated. (Abbiw et al., 1990).

Table 1: List of plants with part and its uses.

S. No.	Constituents	Parts	Uses
01	*Aloe barbadensia*	Leaf	Promote hair growth or conditioner
02	*Trigonella foencum*	Seeds	Promote thick and healthy hair
03	*Phyllanthus emblica*	Fruits	Hair darkening.
04	*Azadirachta indica*	Leaves	Cleansing and anti fungal property
05	*Sapindus mukorossi*	Nuts	Shining property
06	*Acacia concinna*	Fruits pods	Anti dandruff agent
07	*Lawsonia inermis*	Leaves	Improve hair texture
08	*Hibiscus rosea*	Leaves and flowers	Hair conditioner

Evaluation of Herbal Shampoo

To evaluate the quality of prepared formulation, several quality control test including visual appearance, physiochemical control, conditioning performance etc test were performed.

Physical appearance/visual inspection

The formulations prepared were evaluated in terms of their clarity, with brown colour and good in odour with foam producing ability and fluidity.

Determination of pH

The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C with help of pH meter and Litmus paper.

Determine percent of solids contents

A clean dry evaporating dish was weighed and with adding 4 grams of shampoo to the evaporating dish and both was weighed. The correct weight of the shampoo was calculated solely and place the evaporating dish with shampoo was placed on the recent plate once the liquid portion was gaseous. The weight of the shampoo only (solids content) after drying was calculated accurately.

Rheological evaluations

The viscosity of the shampoos was calculated by using Oswalt's viscometer.

Dirt dispersion

In the large test tube have 10 ml of distilled water with adding two drops of shampoo. In this add one drop of India ink and test tube covered with stopper and shake with it ten times. The amount of ink in the foam was measured with as None, Light, Moderate, or Heavy.

Cleaning action

5 grams of wool yarn were placed with grease and it was placed in 200 ml. of water containing one gram of shampoo in an exceedingly flask. Temperature of water was maintained at 35^0C checked with the help of thermometer. The flask was shaked continuously for 4 minutes at maximum the rate of 50 times in a minute. The solution was safely removed and sample was taken out, dried and weighed. The amount of grease removed was calculated by using the following equation of detergency power,

Detergency Power (DP) = 100(1-T/C)

Where 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.

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 affected with grease. The data calculated by following equation given bellow:

$$R_3 = (W_3\text{-}W_1)\, n_1 / (W_2\text{-}W_1)\, n_2 * R_1$$

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. (Ireland et al., 2007)

Detergency ability

The Thompson technique was accustomed judge the usefulness 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 dissolving 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 gaseous at room temperature and their sebum content finded. 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(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 sample. (Ireland et al., 2007)

Foaming ability and foam stability

Cylinder shake method was used for determining foaming ability. 50ml of the 1% shampoo solution was put into a 250 ml graduated cylinder and lined 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 with immediately after shaking the volume of foam at 1 minute intervals for 4 minutes were recorded. (Ireland et al., 2007)

Stability studies

The thermal stability of formulations was studied by placing in glass tubes and they were placed in a humidity chamber at 45°Cand 75% relative humidity. Their appearance and physical stability were inspected for a period of 3 months at interval of one month. (Hadkar et al., 2009)

RESULT AND DISCUSSION

Evaluation of Herbal Shampoos

Physical Appearance/Visual Inspection

The results of visual inspection of series of formulations are listed in table 3. As can be seen, all formulations had the good characteristics and phytochemical constituents as listed in table 3. (Hadkar et al., 2009)

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 follower 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 3, all the shampoos were acid balanced and were ranged 5.5 to 5.9, which is near to the skin pH. (Eldridge et al., 1997)

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 20-29%. As a result, they were easy to wash out.

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 5 shows the goodness of fitting indices for Newtonian, plastic and pseudo plastic flow behaviors. (Gaud et al., 2001)

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 four shampoos showed similar results. These results indicate that no dirt would stays in the foam; so prepared and marketed formulations are satisfactory.

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 remove11. 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 6.

Surface tension measurement

It has been mentioned that a proper shampoo should be able to decrease the surface tension of pure water to about40 dynes/cm12. Surface tension reduction is one of the mechanisms implicated in detergency. The reduction in surface tension of water from 72.8 dynes/cm to 34.70dynes/ cm by the herbal shampoos is an indication of their good detergent action. The results are shown in table 6. (Gaud et al., 2001)

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 MF1, MF2 being a frequent-use cleanser, was expected to have the maximum detergency. Shampoos F1 and F2 also showed moderate detergency. The results are presented in table 6. (Mainkar et al., 2000)

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 four shampoos showed similar foaming characteristics in distilled water. All four shampoos showed comparable foaming properties. The foam stability of herbal shampoos is listed in table 7. 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. (Aghel et al., 2007) (Griffin et al., 1977)

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 8. (Mahran et al., 1996)

Figure 1: Images of herbal shampoos with herbal plant.

Table 2: Formulation of shampoo.

S. No.	Particulars	Part used	Quantity (10ml)
1.	*Aloe barbadensia*	Leaf	0.25gms
2.	*Trigonella foencum*	Seeds	0.25gms
3.	*Phyllanthus emblica*	Fruits	0.25gms
4.	*Azadirachta indica*	Leaves	0.25gms
5.	*Sapindus mukorossi*	Nuts	0.25gms
6.	*Acacia concinna*	Fruits pods	0.25gms
7.	*Lawsonia inermis*	Leaves	0.25gms
8.	*Hibiscus rosea*	Leaves and flowers	0.25gms
9.	Sodium lauryl sulphate	-	2.0 gms
10.	Water	-	q.s.
11.	Perfume	-	q.s.

Table 3: Evaluation of Formulation for physical appearance, pH and Solids.

S. no	Formulation	Physical appearance	pH	Solid
1.	F1	Dark brown, good foaming	5.40	20.91
2	F2	Dark brown, good foaming	5.12	21.66
3	MF1	Dark brown, good foaming	5.86	25.11
4	MF2	Dark brown, good foaming	5.84	27.42

Table 4: General tests of Phytochemical Constituents.

Sr. No.	Test	Observation	Inference
1.	Test for carbohydrates Extract + alpha-naphthol solution in alcohol+ conc. H_2SO_4 from sides of the test tube.	Violet ring is formed at upper at the solution	Positive
2	Tests for protein Extract+ 1% NaOH + Cu(II)SO_4	Purple color produce	Positive
3	Tests for alkaloids Extract + Hager's reagent	Yellow precipitate	Positive
4	Tests for glycosides Hydrolyze small portion of the extract with dilute HCl+1 ml pyridine + 1 ml sodium nitroprusside.	Pink to red color appears	Positive
5	Tests for saponins Dilute 1 ml of extract separately with distilled water to 20 ml and shake in a graduated cylinder for 15 mins	Foam produce	Positive

Table 5: Viscosities of herbal shampoos.

S. no	Formulation	Viscosity
1.	F1	1.19
2	F2	1.16
3	MF1	1.18
4	MF2	1.25

Table 6: Evaluation of Formulation for Cleansing, Surface tension and Detergency.

S. No.	Formulation	Cleaning (%)	Surface tension (dynes/cm)	Detergency (%)
1	F1	24.81	32.28	69.38
2	F2	31.68	31.34	66.28
3	MF1	31.46	32.68	65.59
4	MF2	29.56	31.48	65.28

Table 7: Foam stability of herbal shampoos.

	Foam volume (ml)			
	F1	F2	MS1	MS2
1	178	176	171	174
2	176	174	173	173
3	169	179	178	179
4	168	178	179	171
5	170	174	176	174

Table 8: Stability studies herbal formulations.

Parameters	1month	2month	3month
Physical appearance/visual inspection	Clear	Clear	Clear
pH	5.26	5.24	5.21
Solids contents (%)	22.32	23.45	24.84
Surface tension measurement (dy. /cm)	32.78	31.68	34.72
Rheological evaluations (cps)	1.22	1.24	1.28
Detergency ability (%)	64.36	66.38	60.86
Foaming ability and foam stability (ml)	164	175	169

CONCLUSION

The formulation and development of herbal shampoos were not solely safer than the chemical acquisition agents, however additionally greatly scale back the hair loss throughout hair dressing also as strengthen the hair growth. The hydrogen ion concentration or pH of the shampoos was adjusted to 5, to retain the acidic mantle of scalp. Synthetic preservatives have generally been the explanation for adverse effects like irritation to skin among customers. We have used the physicochemical approach to preservation and by developing a self protective shampoo, have avoided this risk posed by chemical or synthetic 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 merchandise don't fare thus well within the tests conducted by North American nation, they enjoy market popularity, especially if they foam well. This is in the main because of the false notion among customers that 'a shampoo that foams well, works well', and no real effort on the part of manufacturers to counter this fallacy. In the gift state of affairs, it seems improbable that herbal shampoo, although better in performance, better in stability and safer than the synthetic ones, will be popular with the consumers.

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