Academic Sciences International Journal of Pharmacy and Pharmaceutical Sciences

ISSN- 0975-1491 Vol 5, Suppl 3, 2013

Research Article

FORMULATION AND EVALUATION OF HERBAL SHAMPOO HAVING ANTIMICROBIAL POTENTIAL

NAMITA[1], NIMISHA[*1]

Amity Institute of Pharmacy, Amity University Uttar Pradesh Lucknow. Email: Nsrivastava3@amity.edu Nimisha4u31@gmail.com

Received: 24 Jun 2013, Revised and Accepted: 30 July 2013

ABSTRACT

Objective: The aim of present research work is to develop a herbal shampoo for hair growing and strengthing without affecting or damaging hair. Reetha, Amla, Neem, Bringraj, Jatamanasi & Aloe vera herbs have been selected on the basis of a traditional system and scientific justification with modern uses.

Material and Methods: Hair formulation of *Sapindus trifoliatus* (sapindaceae), *Phyllanthus emblica* (phyllanthaceae), *Azadirachta indica* (Meliaceae), *Eclipta alba* (Asteraceae), *Nardostachys jatamansi* (Valerianaceae) and *Aloe vera* in two concentrations (1and 1.5%) in the form of herbal shampoo were evaluated and studied for hair washing and conditioning activity.

Evaluation: The pharmacognostical standardization has been done as per the, The Ayurvedic Pharmacopoeia (Volume I 1989, Volume II 1999, Volume III 2001) of India (API).It includes; for Sapindus trifoliatus, foreign organic matter (0.98%), water soluble extractive (78.92%), total ash (8.20 %), acid insoluble ash (0.39%), pH (4.53) and moisture (7.77%). All the values are in compliance with API part 1 vol-2. The results revealed that the hair growth activity of each drug was found proportional to the concentration range tested. The formulation containing 1% of each drug used for the study showed excellent results.(foam value 350mm). Excellent results of washing and conditioning were seen in formulation prepared by aqueous extraction technique. Accelerated stability testing of two final sample has been conducted in the environmental chamber with temperature 25 ± ^{10}C and humidity 60 ± 10% RH. All the products were found to be stable with no sign of phase separation and no change in the color. The patch test for sensitivity testing has also been done and no evidence of skin irritation and allergic sensitization.

Conclusion: Herbal shampoo will not only give hair protection but also conditioning effect, shine and manageability

Keywords: Hair formulation, Herbal shampoo, Aqueous Extraction.

INTRODUCTION

The hair of the head has historically been associated with beauty and social distinction. The hair has been trimmed, shaped, and even colored since the most ancient times, relatively little emphasis has been placed on the process of cleaning it. Real technology in the cleaning of hair and scalp has developed only in this century. First came the mass distribution of cake soap and sanitary facilities to make bodily cleanliness and personal hygiene practical. Next came the specialization of branded shampoo products for the hair and scalp, offered in a multiplicity of types and forms.[1]

Hair care by itself can induce a state of self confidence and may reflect social status. This may explain significant differences in shampooing regimens, which range from once or twice a week to once a day.

Hair is a mid way between nature and culture. Hair care attitudes are different from one society to another regardless of economic differences, and from one person to another within societies.[2]

Harry defined shampoo as "a preparation of a surfactant i.e surface active material in a suitable form – liquid, solid, powder. But the usage of surface active material becomes very harmful from long time for the youth as well as our environment. Various synthetic compounds, chemicals, dye and their derivative has been proved to cause various skin diseases having numerous side effects. The word herbal is a symbol of safety in contrast to the synthetic one which has adverse effects on human health. Thus there is increasing attractiveness of herbal cosmetics and the tremendous range of herbal products now generally available to the public.[3]

Now-a-days the usefulness of herbs in the cosmeceutical production has been extensively increased in personal care system and there is a great demand for the herbal cosmetics.

The basic idea of hair growth enhancing & conditioning shampoo lies deep in the Rigveda, Yajurveda, Ayurveda, Unani and Homeopathic system of medicine. These are the products in which herbs are used in crude or extract form. These herbs should have varieties of properties like nervine tonic, cleansing and softening activity, antiseptic properties, promote the growth of hair, and antibacterial etc.[4]

Today's busy life schedule has created the negligence of an individual to protect their hair from various problems. People don't have time for different treatment for getting good results.

The objective of this study was to develop a method of for hair growing and strengthing without affecting or damaging hair. For this herbal drugs were use for the formulation of shampoo.

Shampoo is a polyherbal formulation that consist of extracts of of *Sapindus trifoliatus* (sapindaceae) common name Reetha, *Phyllanthus emblica* (phyllanthaceae) common name Amla, *Azadirachta indica* (Meliaceae) common name Neem, *Eclipta alba* (Asteraceae) common name Bhringraj, *Nardostachys jatamansi* (Valerianaceae) common name Jatamanasi and Aloe vera gel. These herbs have been selected on the basis of a traditional system and scientific justification with modern uses.[5]

Reetha is used as the main ingredient in soaps and shampoos for washing hair, as it is considered good for the health of hair. The herb is also used in the treatment of extra salvation, migraine, epilepsy and chlorosis., as it has gentle insecticidal properties. The plant is known for its antimicrobial properties that are beneficial for septic systems.[6]

Amla is used as cosmetic in India. It is an accepted hair tonic in traditional recipes for enriching hair growth and pigmentation.

Extract from the leaves of Neem were first used in India to treat fungal infection, and skin diseases. It has also been used from centuries as anti inflammatory, antifungal, antibacterial, anti tumor activities.[7]

Bhringraj is the main herb for the hair care in Ayurveda. It is believed to maintain and rejuvenate hair, teeth, bones, memory, sight, and hearing.

Jatamansi has a rich history of medicinal use and has been valued for centuries in Ayurvedic (Indian) and Unani (ancient Greco-Arab) systems of medicine. It shows antifungal activity.[8]

MATERIALS AND METHODS

All the drugs and excipients were collected from Bacfo Pharmaceuticals (India) Ltd, Limited C-15, sector- 2, Noida.

Pharmacognostical standardization

Quantitative standards of all the drug components were carried out as per The Ayurvedic pharmacopoeia of India (API) methods and compared with API standards. (Table 1)

Method of Extraction

All the drugs were weighed accurately & aqueous extraction had been done (10 times of the weight of the drug i.e. 5g in 50ml of water on water bath at 80-100°C). As the solution concentrated up to 20 ml, filtration was done. Residue had been taken & volume was making up to 40ml, again was boiled. After remaining 20 ml was filtered & the same procedure was followed again.

Drug Formulation

The formulation components used were listed in Table 2. The hair formulations of 1 and 2% of Sapindus trifoliatus, Phyllanthus emblica, Azadirachta indica, Eclipta alba, Nardostachys jatamansi and Aloe vera were prepared by cloth pouch method. Component A (water soluble) consisting of drug extract, PEG 400 & Aloe vera gel and components B (oil soluble) i.e. SLS (Sodium lauryl sulphate), CAPB (Cocoamido propyl betain), & Polyquaternium-7 were heated separately to 60- 80°C & stir until becomes homogeneous. Added component A to component B with slow agitation without production of foam. Saturated solution of sodium chloride was added to increase viscosity. Cool with stirring & perfume was added at 45 ± 5 °C. [9, 10] (Table no 2)

Table 1: Quantitative Standards of Selected Herbs

Drugs/Parameters	Reetha *Sapindus trifoliatus*	Amla *Phyllanthus emblica*	Neem *Azadirachta indica*	Bhringraj *Eclipta alba*	Jatamansi *Nardostachys jatamansi*
Foreign matter % w/w	0.98	1.04	0.70	1.03	2.81
pH	4.12	3.91	6.65	6.79	5.43
Moisture %	7.56	8.98	10.95	9.31	8.16
Water soluble extractive %w/v	78.92	57.60	18.87	16.56	6.86
Alcohol soluble extractive %w/v	-	42.85	8.08	5.99	3.00
Total Ash %w/w	8.20	6.14	5.88	14.33	8.04
Acid Insoluble Ash %w/w	0.39	0. 61	0.58	7.90	3.33
Reference (compliance with)	In house	API Part I Vol I	API Part I Vol II	API Part I Vol II	API Part I Vol I

Table 2: Composition of Herbal shampoo

S. No.	Solution	Drugs	Quantity taken for F1	Quantity taken for F2
1.		Extract	21.0 ml	19.00 ml
2.	A	Poly ethylene glycol- 400 (PEG-400)	2.00g	2.50g
3.		Water	qs	qs
4.		Aloe vera	2.00g	2.00g
5.		Polyquaternium-7	6.25g	6.33g
6.		Sodium lauryl sulphate (SLS)	25.00g	25.40g
7.	B	Cocoamido propyl betain (CAPB)	12.50g	13.10g
8.		Sodium chloride	1.00g	1.00g

Evaluation of Shampoo

The prepared formulations were evaluated using standard methods of general evaluation, organoleptic evaluation, microbiological evaluation and chemical evaluation including specific gravity, pH, detergent content, solid content, viscosity, surface tension. (Table no.3, 4 & 5)

1) Determination of Active Detergent Content

Sample of sufficient size was weighed accurately to give approximately 0.32g of combined SO3 in to 250ml beaker. Sample size was crucial. 700 to 800ml of warm water was transferred quantitatively to a 1ltr volumetric flask. Warmed on steam bath & shaken gently until the sample was dissolved & solution was clear. Cool, diluted to the mark & mixed thoroughly.

10ml of the sample solution was pipette out in to 100ml glass stopper cylinder (25x 300mm). 25 ± 0.5ml of ethylene blue solution & 10±0.5ml chloroform was added. Titrate with shaking the cylinder carefully after each addition (to avoid emulsion) & maintaining temperature with in prescribed limit of 20-30° C by immersion in water bath. As the endpoint is approached, the rate of transfer of colour had been increased.

If the appropriate titration volume of A is known to 80% of the required titrating solution should be added before shaking since this avoids emulsion formation. Application of vacuum to titration cylinder may help to break some emulsion, if formed. The end point is reached when both layers have some colour intensity.

The end point is very sharp & 0.5ml will cause a distinct change in colour distribution at or near the equivalence point.

Cationic Solution (Solution A)

Weigh 1.5 ± 0.001g of cetyl trimethyl ammonium bromide in to 250 ml beaker. Add 100ml of distilled water & stir until dissolved. Transfer quantitatively to 1 ltr volumetric flask & make to volume. Mix properly & standardized against solution B.

Anionic solution (Solution B)

Weighed accurately such amount of standard alkyl sulphate of known combined SO3 or active content so as to give exactly 0.32g Combined SO3 in a 250ml beaker and was dissolved in 100 to 200g of warm water. Quantitatively transferred to a 1 ltr volumetric flask & made to volume with water at room temperature, mixed thoroughly. This was the primary standard against which solution A was standardized. Solution B was 0.004 N.

Calculation

$$\% \text{ combined } SO3 = V \times N \times 800 / M$$

Where,

V= Vol in ml of solution A used in the titration

N-= normality of solution A

M= mass in gm of sample in the aliquot.

% Active detergent content

Persent combined SO3 = mol mass of active detergent x 10/ 100

2) Determination of Foam Height

While the shampoo solution was ageing, circulate the water at 30 ± 2°C through the water jacket of the receiver so as to bring it to the proper temperature. Rinse down the wall of the receiver with DW & as an indication of cleanliness; observe whether the water drains down the walls in an unbroken film. At the completion of the ageing period close the stop cork of the bottom of the receiver. Rinse the wall of the receiver with 50ml of solution, using a pipette & after draining to the bottom of the receiver adjust the stop cork so that the level of the solution in the receiver is exactly at the 50ml mark; using the slight suction for the purpose. Immediately place it in a position at the top of receiver & open the stop cork.

When all the solution has run out of the pipette start the stop-watch take a reading of the foam height & take the second reading at the end of 5min.Take the reading by measuring the foam production at the top of the foam column at the highest average height to which the rim of the foam has reached. This height is proportional to the volume of air remaining in the foam.

3) Determination Of pH

pH was determine at the temperature of 27°C +_ 2°C. In the case of liquid shampoo, pH was read directly in the sample in the pH meter.

4) Microbiological examination of shampoo

Media & buffer

A.) Soybean casein digest agar medium

40gm media was dissolved in 1000ml of distilled water. Gently heated to dissolve the medium completely and sterilized by autoclaving at 15 psi (121 °C) for 15min.

B.) Stock solution ph 7.2 phosphate buffer

34gm media was dissolved of monobasic phosphate buffer in about 500ml of water contained in 500ml volumetric flask. pH was adjusted to 7.2 ± 0.1 by the addition of sodium hydroxide solution (4%). Water was added to volume & mixed. Sterilize at 122 °C for 20min was stored under refrigeration.

C.) Diluted phosphate buffer solution ph 7.2

1ml was diluted of stock solution with distilled water in the ratio of 1: 800. 50ml in each conical flasks was filled & sterilize at 122 °C for 20min.

Procedure

Melt sufficient number of soyabean casein digest agar medium tubes in a hot water bath & transfer while hot in to a constant temperature. Water bath maintained at 48 ± 2°C.

1g of the sample was weighed & transferred aseptically of the sample to the conical flask containing sterile 50ml of dil. Phosphate buffer at pH 7.2. Shake well. Pipette Out in 1ml portion in to 3 sterile Petri dishes. Pour melted & cooled (at 45°C) soyabean casein digest agar over it, & rotate the plate to mix thoroughly. Incubate the plates at 32°C for 74 hrs in an inverted portion.

Determine the average no. of colonies on soyabean casein digest agar medium & multiply by 30 the dilution factor. If no. of colonies is recovered from any of the plate it can be started as less than 50 microorganisms per gram.

Determine percent of solid 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 on the hot plate until the liquid portion was evaporated. The weight of the shampoo only (solids) after drying was calculated.

Rheological evaluations

The viscosity of the shampoos was determined by using Viscometer. The viscosity of the shampoos was measured with the temperature and sample container's size was kept constants during the study.

Surface tension measurement

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

R2 = (W3-W1) N1 x R 1 (W2 -W1) N 2

Where W 1 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.

Accelerated stability testing

Accelerated stability testing of prepared formulations i.e. F1andF2 were conducted at 40 ± 2°C temperature and 75± 5% relative humidity and studied for 90 days. (Table no.6)

RESULT AND DISCUSSION

Medicinal plants used in the formulation of herbal shampoo were found as rich source of novel drugs. These plants were reetha, amla, neem, jatamansi, and bhringraj had been reported for hair growth and conditioning. The various quality control parameters like viscosity, pH, detergent content, foam height were checked. All parameter gives favorable result. The result obtained on present study shows that the active ingredients of these drugs when incorporated in shampoo gives more stable products with good aesthetic appeal.

Physical appearance: Both formulations were found to be semi liquid in nature, have uniform texture and characteristic odour. (Table No. 4)

pH value: The pH of the shampoo was found to be in range of 6-7 which shows no harmful effect on scalp and hair. Both the formulations were shown pH nearer to skin required. (Table No. 3)

The results of Detergent content, Foam value and specific gravity (wt/ml) of both formulations showed satisfactorily value. (Table No. 3)

Thermal stability: At temperature 40 ± 2°C and relative humidity 75± 5% for 90 days. Formulation was found to be stable (Table No. 3)

Degradation of product: No degradation of product was found at 40 ± 2°C temperature and 75± 5% relative humidity (Table No.3)

Microbial count (cfu/gm): The result of total aerobic microbial count, mould and yeast count were presented in table and showed satisfactorily values. (Table No. 5, Fig No. 1)

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. (Table No.3)

Rheological evaluations

These formulations showed pseudo plastic behavior which is a desirable attribute in shampoos formulation. 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. (Table No. 3)

Surface tension measurement

Surface tension reduction is one of the mechanisms implicated in detergency. The reduction in surface tension of water from 72.8 dynes/cm to 28.76 dynes/ cm by the herbal shampoos is an indication of their good detergent action. (Table No. 3)

Accelerated stability study: Accelerated stability testing of prepared formulations F1and F2 were conducted at 40 ± 2°C temperature and 75± 5% relative humidity and studied for 90 days. Excellent results were obtained. [11, 12] (Table No. 6)

Table 3: General evaluation of herbal shampoo

S. No.	Test	Formulation	
		F1	F2
1	pH value	6.78	6.80
2	Foam value (mm)	350	348
3	Specific gravity (wt/ml)	1.116g/ml	1.115 g/ml
4	Thermal stability	Ok	Ok
5	Solid Content(%)	22.11	23.12
6	Viscosity (cps)	67.00	66.76
7	Surface Tension (dynes/cm)	28.76	29.00
8	Detergent content	6.035	6.035
9	Degradation of product	Nil	Nil

Table 4: Organoleptic Evaluation of herbal shampoo

S. No.	Specifications	Formulation	
		F1	F2
1	Physical appearance	Semi liquid	Semi liquid
2	Texture	Ok	Ok
3	Colour	Brown	Brown
4	Odour	Characterstic	Charecterstic

Table 5: Microbiological Evaluation of herbal shampoo

S. No.	Tests	Formulation	
		F1	F2
1	Total aerobic microbial count	$0x10^2$	$1x10^2$
2	Mould and yeast count	$0x10^2$	$0x10^2$

Table 6: Accelerated Stability Testing For Herbal Shampoo

Months/ Tests	Herbal shampoo (1%)				Herbal shampoo (1.5%)			
	Initial month	After – 1 month	After – 2 month	After – 3 month	Initial month	After – 1 month	After – 2 month	After – 3 month
Physical appearance	liquid	liquid	liquid	Liquid	liquid	liquid	liquid	liquid
Texture	Ok	Ok	Ok	Ok	Ok	Ok	Ok	Ok
Color	Dark brown	Dark brown	Dark brown	Dark brown	Dark brown	Dark brown	Dark brown	Dark brown
Odour	Charecteristic	Charecteristic	Charecteristic	Charecteristic	Charecteristic	Charecteristic	Charecteristic	Charecteristic
pH value	6.78	6.79	6.68	6.68	6.92	6.99	6.88	6.78
Foam value	350mm	344mm	349mm	350mm	340mm	344mm	342mm	345mm
Specific gravity (wt/ml)	1.116g/ml	1.118g/ml	1.101g/ml	1.119g/ml	1.131g/ml	1.134g/ml	1.138g/ml	1.133g/ml
Thermal stability	Ok	Ok	Ok	Ok	Ok	Ok	Ok	Ok
Detergent content	6.035	6.035	6.035	6.035	6.035	6.035	6.035	6.035
Degradation of product	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Microbial count(cfu/gm)	$0x10^2$ $0x10^2$	$0x10^2$ $0x10^2$	$0x10^2$ $0x10^2$	$0x10^2$ $0x10^2$	$0x10^2$ $0x10^2$	$0x10^2$ $0x10^2$	$0x10^2$ $1x10^2$	$1x10^2$ $0x10^2$

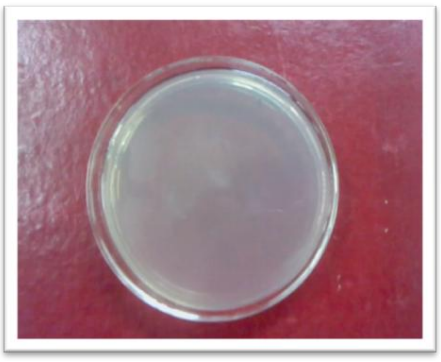

Fig. 1: Photograph showing microbial count

CONCLUSION

A survey of global hair care market trends indicates that consumer use of herbal hair products has significantly increased over the past years. Because hair shampoo are known to damage the hair cuticle and leave brittle, dull and dry hair. The factors like UV radiations, use of harsh chemical products have direct and indirect impact on to the hair.

To overcome this entire problem the present study has the best undertaken to design a herbal shampoo which will not only give hair protection but also conditioning effect, shine and manageability. The present work focuses on the potential of herbal extracts from cosmetic purposes.

ACKNOWLEDGEMENT

Authors thank BACFO Pharmaceuticals (India) Ltd and Amity Institute of Pharmacy, Amity University Uttar Pradesh, Lucknow Campus for providing the necessary facilities for the experimental work

REFERENCES

1. Barel AO, Maibach HI, Paye M. Handbook of Cosmetic Science and Technology. New York: Marcel Dekker, Inc; 2001.p.575
2. Sagarin E, Balsam MS, Cosmetics Science and Technology. Wiley: India Edition; 2 ed (Vol2).p.73-75.
3. Zhang L, Demain AL, Natural Products Drug Discovery and Therapeutic Medicine. Totowa, NJ:Human Press; 2005.p.3-5.
4. Evans WC, Trease GE, Pharmacognosy. UK: Elsevier Saunders; 1996(15 th ed). p.5.
5. Maithani Alok, *Azadirachta indica* (neem) leaf: a review. Journal of Pharmacy Research, 2011. Vol. 4(6), 1824-1827.
6. Kausik Biswas, Ishita Chattopadhyay, Ranajit K. Banerjee and Uday Bandyopadhyay, Biological activities and medicinal properties of neem (*Azadirachta indica*). Current Science, 2002.Vol. 82, 1336-1345.
7. Mahajan UN, Wasule DD. Sunscreen and anti-oxidant activities of herbal gel formulations. Pharmacognosy Magzine, 2008. Vol. 13(4), 99-101.
8. Bhaskar G, Arshia S, Priyadarshani SRB. Formulation and evaluation of topical polyherbal antiacne gels containing *Garcinia mangostana* and *Aloe vera*. Pharmacognosy Magzine, 2009. Vol. 19 (5),93-99.
9. Firthouse PU. Effects of *Ocimum sanctum* and *Azadiracta indica* on the formulation of Antidandruff Herbal Shampoo Powder. Der Pharmacia Lettre, 2009. Vol. 1(2), 68-76.
10. Mainkar A R, Jolly C. Formulation of natural shampoos. Int J Cosmet Sci, 2001. Vol. 23(1), 59 - 62.
11. Pounikar Y, Jain P, Khurana N, Omray L. K. Patil S. Formulation And Characterization Of Aloe Vera Cosmetic Herbal Hydrogel. Int J Pharmacy Pharm Sci, 2012. Vol. 4(4), 85-86.
12. Adriana D, Marcos R, Eugenia A, Alicia S R. Topical Antiinflammatoryactivity Of Tripodanthus Acutifolius Flowers Gel Formulation.J Pharmacy Pharm Sci, 2012. Vol. 4(1), 183-186.