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Original Article

"USE OF TRADITIONAL INDIAN HERBS FOR THE FORMULATION OF SHAMPOO AND THEIR COMPARATIVE ANALYSIS"

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ABSTRACT

Objective: The aim of present research work was to formulate herbal shampoo using aqueous extracts of *Aloe vera* (Korpad), *Acacia cancina* (Shikakai), *Sapindus Mukorossi* (Reetha) and *Phyllanthus emblica* (Amla) as a replacement to synthetic surfactants and carry out a comparative analysis for their cleansing activity.

Methods: Herbal shampoo formulation was carried out using different phytoextracts mixed in a definite proportion. Phytochemical screening was carried out to estimate total phenolic, flavonoid and saponin content. CAPB (Cocoamido propyl betain) was used as a conditioner for the formulation. Further, the formulations were analyzed for physicochemical properties such as pH, dirt dispersion, detergency ability and foaming ability which are pre-requisite for shampoo preparations.

Results: It was observed that *Phyllanthus emblica* formulation showed the highest cleaning ability i.e. 96% due to the presence of rich flavonoid content while *Acacia cancina* formulation contributed the highest detergency ability i.e. 94.96% due to the presence of rich saponin content. All the formulation showed high foaming ability and stability which is a prerequisite of shampoo preparations.

Conclusion: The results obtained during experimentation clearly indicate a promising formulation of quality enhanced herbal shampoo with a unique aroma, colour and potential for cleaning and foaming ability.

Keywords: Herbal shampoo, Antioxidants, Reetha (Sapindus Mukorossi), Shikakai (Acacia cancina), Amla (Phyllanthus emblica), Korpad (Aloe vera), CAPB, Foam stability

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INTRODUCTION

Shampoo plays an important role in the removal of surface grease and dirt from the hair shaft and scalp. The primary role of shampoo is to carry out cleansing or detergent action. However, the foaming characteristic of shampoo plays a significant role in its acceptability [1]. The shampoo sector is probably the largest scale amongst hair care products since they are one of the important cosmetic products used in daily life. Hair cleansers play an important role not only in cleansing but also in imparting glossy nature to hair [2]. With the emerging role of ayurveda, several herbs and florals have been identified for their impacting role in the cosmetic industry. Herbal cosmetics like face washes, conditioners, soaps, etc. have been dominating this industry [3]. Herbs were used as food rather medicine till the present date as they are natural and fulfill the nutrient requirement of human individuals. But several herbal extracts have proved themselves fruitful for their utilization in curing diseases related to scalp layer [4-6].

The word herbal signifies a symbol of safety in contrast to the synthetic one which has adverse effects on human health [7]. Today's busy life schedule has created the negligence of an individual to protect their hair and maintain the hair homeostasis [8]. So there is a need to derive formulations for avoiding hair damages that contain herbal extracts. It has been reported that aqueous extracts of Neem (*Azadirachta indica*), *Acacia cancina*, *Sapindus Mukorossi*, Tulsi (*Ocimum sanctum*), *Aloe vera*, lemongrass, etc. have proved major role in the removal of dandruff, hair glossing and treatment of hair scalp problems since ancient times [9,10]. These aqueous extracts of herbs can be used to formulate hair cleansing solutions [11].

The shampoo is formulated with non-ionic, cationic or anionic agents who act as surfactants [12]. Sodium lauryl sulfate based detergents are the most common, but the concentration will vary considerably from brand to brand and even a manufacturer product range. Cheap shampoos may contain a high detergent concentration

while expensive shampoos may contain a very little of a cheap detergent [13, 14]. The most popular conditioning agents used in shampoos today are cationic polymers and surfactants, some of which may present the risk of nitrosamine development. Nitrosamines are known to be hazardous to health. Formaldehyde or formaldehyde based chemicals may be used as preservatives in the formulation, which may cause adverse effects such as skin irritation or sensitization [15]. So a formulation using natural extracts, as surfactants is a need for today.

Sapindus mukorossi has been used as a herb for treatment of extra salvation, migraine, epilepsy and chlorosis. It also shows insectkilling properties. The plant is known for its antimicrobial properties that are beneficial for septic systems [16]. It has been proved that *Sapindus Mukorossi* shows hepatoprotective and antioxidant property due to the presence of phenolic and flavonoid compounds [17]. *Phyllanthus emblica* is rich in ascorbic acid and polyphenols. It is observed that *Phyllanthus emblica* extracts have prominent antioxidant potential. They have also been suggested in the diet since long time [18]. *Phyllanthus emblica*) is also called as rejuvenating medicine due to its multiple health benefits and heuristic medicinal value. It shows anti-inflammatory and antigenotoxic activity [5, 19]. It is also used as a cosmetic in India. It is an accepted hair tonic in traditional recipes for enriching hair growth and pigmentation [20].

Aloe vera is used in herbal medicine since a long time. *Aloe vera* is widely used in cosmetics and alternative medicine industries, being marketed as variously having rejuvenating, healing, or soothing properties [7]. *Acacia cancina* is used in herbal preparations from ancient time for hair growth and cleaning [1].

The objective of this study was to develop a formulation for hair growing and strengthening without damaging. For this process, herbal extracts of plants like *Phyllanthus emblica, Acacia cancina, Sapindus Mukorossi* and *Aloe vera* were used, and the formulation of shampoo was carried out. CAPB was used as a conditioner for the

preparation [8]. Prepared shampoos were comparatively evaluated for pH, percent solid content, detergency ability, dirt dispersion and cleaning action test [15, 21]. Foaming ability and foam stability were also checked for the herbal shampoos. Phytochemical screening for total flavonoid, phenolic and saponin content was carried out.

The present century has many herbal shampoos available in the market which contains herbal ingredients such as plant extracts and essential oil [22]. There are large numbers of plants which are reported to have beneficial on hair and are commonly used in shampoos [23]. Herbal drugs or their formulations will henceforth become an alternative to the synthetic drugs. But there is a need for formulating herbal shampoos which would have better cleaning and conditioning activity.

MATERIALS AND METHODS

Materials

All the plant materials were obtained from a local botanical garden in Pune and were identified and authenticated by college botanist. CAPB as a conditioner and other chemicals were purchased from Sigma-Aldrich.

Methods of plant extract preparation

5 g of plant extract was added to 50 ml (10% w/v) and the mixture was heated at $100 \degree$ C for 10 min. When the solution concentrated to 20 ml, the residue was separated, and volume was made upto 40 ml by adding distilled water. The mixture was boiled again and filtered [16].

Formulations of herbal extract shampoo

The plant extracts were mixed in proportion mentioned in table no. 1. The solutions I & II was prepared separately to avoid coagulation and later mixed together. The solution I consists of herbal extract, PEG (polyethylene glycol), SLS (sodium lauryl sulphate), polyquaternium 7 and distilled water. Solution II consists of CAPB (coco amido propyl betain) and sodium chloride was added for saturation to increase its viscosity and conditioning effect. The solution I was heated at 80 °C for 10 to 15 min till a clear homogenous solution was obtained, and it was added to solution II after cooling [20, 24].

Table 1: Ingredients used in formulation of shampoo

S. No.	Solution	Chemicals	Quantity (100 ml)
1.	Ι	Extract [Aloe vera, Acacia cancina, Phyllanthus emblica & Sapindus Mukorossi]	21.00 ml
2		PEG (Polyethylene Glycol)	2.00 g
3		Polyquaternium 7	6.25 g
4		SLS (Sodium Lauryl Sulphate)	25.00 g
5		Distilled water	79.00 ml
6	II	CAPB	12.50 g
7		Sodium Chloride	1.0 g

Physical appearance and content analysis

For the evaluation of herbal shampoo, it was assessed using several quality control tests such as pH, physical appearance and determination of solid contents.

Determination of pH and physical appearance

The pH of 10% shampoo solution in distilled water was determined at room temperature using a pH meter, and its physical appearance was noted [3].

Determination of percent solid content

The clean dry evaporating dish was weighed, and 4 gms of shampoo was added to the evaporating dish. The dish with shampoo content 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 shampoo (residual mass) after drying was calculated [25].

Phytochemical screening

Estimation of total flavonoids content by Aluminum chloride method

Total flavonoid content was estimated by spectrophotometric analysis. In a test tube, 0.3 ml of extracts, 3.4 ml of 30% ethanol, 0.15 ml of Sodium nitrate (NaNO₃) (0.5 M) and 0.15 ml of Aluminium chloride (AlCl₃) were mixed. After 10 min of incubation, 1 ml of 1M Sodium Hydroxide (NaOH) was added. The samples were properly mixed and absorbance was measured at 506 nm. The standard curve for total flavonoids was prepared using standard quercetin solution with the same procedure. The total flavonoid content was expressed in milligrams of quercetin equivalents per gram of extracts [26-28].

Estimation of total phenolic content

The total phenolic content was determined by the similar spectrophotometric method. 1 ml of sample was mixed with 1 ml of Folin Ciocalteu's phenol reagent. After 5 min of incubation, 10 ml of 7% Sodium carbonate solution (Na_2CO_3) was added to the mixture. The volume was made up to 25 ml by addition of deionized distilled

water, and it was thoroughly mixed. The sample mixtures were kept in dark at 90 °C at room temperature after which absorbance was measured at 750 nm. A standard curve for total phenolic content was prepared using gallic acid standard solution. The total phenolic content was expressed in milligrams of gallic acid equivalents per gram of sample [29, 30].

Estimation of total saponin content

Saponins content was measured with the vanillin–perchloric acid colorimetric method. A volume of 0.05 ml of the extract was mixed with 0.1 ml 5% vanillin-glacial acetic acid solution and to this mixture 0.4 ml perchloric acid was added. The tube containing the mixture was vortex stirred to ensure uniform distribution and then it was transferred to a water bath at 70 °C for 15 min, and then removed and placed in ice-water to cool. Following this, 2.5 ml glacial acetic acid was added to each tube. Then the solution was mixed well and the absorbance was measured against a freshly prepared reagent blank at 540 nm. A standard calibration plot was generated using known concentrations of Quillaja saponin. Saponin content of the extracts was expressed as milligrams of Quillaja saponin equivalents per 100g dry weight of sample [31].

Physiochemical analysis

The physiochemical analysis was carried out using cleaning action, detergency ability, foamability and foam stability assay to compare the activity of the formulation.

Dirt dispersion test

2 ml of shampoo was added in a large test tube containing 10 ml of distilled water. 0.1 ml of ink was added in the test tube; it was stopped and shaken for ten times. The amount in the foam was estimated as none, light, moderate or heavy [32, 1].

Cleaning action test

5 gm of cotton cloth (wool can also be used as an option) was added with grease and it was placed in 200 ml of water containing 0.5% (w/v) of shampoo in a flask. The temperature of water was maintained at 35 °C. The flask was shaken for 4 min at the rate of 50

shakes per min. The solution was removed, and the sample was taken out, dried & 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 the grease in the control sample, and T is the weight of the grease in the test sample [22].

Detergency ability

The Thompson method was used to evaluate the detergency ability of the samples. Briefly, a crumple of hair was washed with 5% Sodium lauryl sulphate (SLS) solution, then dried and divided into 3g weight groups. The samples were suspended in n-hexane solution containing 10% artificial sebum and the mixture was shaken for 15 min at room temperature and their sebum content determined. In the next step, each part, one washed with 0.1 ml of the 10% test shampoo and 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 will be calculated using the following equation:

DP = 100 (1-T/C) in which,

DP is the percentage of detergency ability. C is the weight of the sebum in the control sample and T is the weight of the sebum in the test sample [15].

Foaming ability and foam stability

Cylinder shake method was used for determining foaming ability. 50 ml of 1% shampoo solution was put into a 1000 ml graduated cylinder and covered the cylinder with the hand and shaken for 10 times were recorded. The total volume of the foam contents after 1 min shaking was assessed. Foam volume was calculated only.

Immediately after shaking the volume of foam at 1-minute interval for were recorded. This process was continued for 5 min and the foam volume was measured [23].

Statistical analysis

Values are shown in tables and fig. is the mean of at least three independent replicates and the results are expressed as mean \pm SD. The data were subjected to one-way analysis of variance (ANOVA), where the differences between groups and standards were determined by T test (p value ≤ 0.05 was regarded as significant).

RESULT AND DISCUSSION

With the help of different aqueous extracts, an herbal shampoo was formulated by mixing different constituents in specific proportions. Selected plant materials are rich in polyphenol compounds such as a flavonoid, phenolic and saponin. They have found to exhibit cleansing and surfactant properties. These herbal extracts also provided viscosity to the formulations. The formulations were analyzed during the initial month of preparation and after the third month to check their stability.

Evaluation of formulation for physical appearance, transparency and odour, pH and percent solids content

It is mandatory for an herbal preparation to have good and appealing appearance. The formulation of natural extracts was evaluated for physical appearance, transparency and odor. The results are mentioned in table 2. There were no significant differences in transparency and odor of formulations. But *Aloe vera* showed appealing purple color as compared to others.

pH plays an important role during the formulation of shampoos since it helps to minimize eyes irritation and enhances hair quality [1]. All the formulations had pH close to 5.5 which is a standard range for commercial shampoos.

Table 2: Physical appearance and content analysis

S. No.	Formulated	Physical appearance	Transparency	Odour	рН	рН	Total solids
	extract				(Initial mo)	(After 3 mo)	content
1.	Aloe vera	Light purple, Good foaming	Opaque	Good	5.5±0.3	5.87±0.2	3.042 g
2.	Acacia cancina	Pale orange, Good foaming	Opaque	Fair	5.5±0.2	5.72±0.2	2.909 g
3.	Sapindus Mukorossi	Faint yellow, Good foaming	Opaque	Good	5.5±0.3	5.70±0.3	2.374 g
4.	Phyllanthus emblica	Faint orange, Good foaming	Opaque	Fair	5.5±0.2	5.70±0.1	3.069 g

Sample size, n=5

The formulations were assessed for pH values after 3 mo when the similar results were observed. It has been suggested that formulation with 30-50% of solids can easily be utilized and washed out. If the lower content of solid is present, then the formulation becomes watery and cannot be applied [20]. The herbal shampoo formulation had higher solid content compared to the standard range which describes their viscous nature and effective applicability.

Phytochemicals screening

The content of polyphenol (flavonoid, phenolic and saponin compounds) was determined using different tests. These polyphenols are reported to exhibit antimicrobial, anti-inflammatory and antidandruff activity. It has also been suggested that saponins content may provide surfactant properties for any extracts [26].

Phytochemical screening suggests the presence of rich phytochemical present in the formulated shampoos. *Phyllanthus emblica* shows the higher content of flavonoids. *Aloe vera* has a higher content of phenolics while saponins are present in higher amounts in *Acacia cancina*.

It has been observed during studies that *Phyllanthus emblica* had a higher concentration of flavonoids i.e. 3.33 ± 0.34 mg/g of QE) compared to other extracts. It suggests that the *Phyllanthus emblica* shampoo formulation will have higher antioxidant potential.

The total phenolics was found to be higher in *Aloe vera* i.e. 3.71 ± 0.27 mg/g of Gallic acid equivalent in comparison to others. Saponin content estimated for *Acacia cancina* was 2.25 ± 0.03 mg/g of quillaja saponin equivalents which was highest as compared to other extracts. It proves that *Acacia cancina* extracts is rich in saponins content which increases in foaming and cleaning ability. *Phyllanthus emblica* had a higher content of flavonoids. It can be suggested that the presence of these flavonoids, phenolics and saponins will provide additional features for surfactant and cleansing property of herbal formulation.

Dirt dispersion, cleaning and detergency ability

Dirt dispersion test was carried out by dispersing ink in shampoo preparations. It is suggested that ink or dirt saturation in foam is difficult for rinsing and gets deposited again on hairs. If ink or dirt stays into water portion, it proves better cleansing activity [26]. All the formulations showed the moderate dispersion of ink in the water portion which shows their actual effectiveness.

Cleansing and detergency abilities are two important aspects for shampoo formulation. Hydrophobic molecules such as phenolics and flavonoids show grease encapsulating i.e. cleansing activity. It was observed that *Phyllanthus emblica* formulation had highest cleansing ability i.e. 90.00% as compared to others due to the higher content of flavonoids. It can also be suggested that polyphenols present in *Phyllanthus emblica* should have contributed to its activity.

Detergency ability depends upon the content of surfactant present in the sample extracts. Saponins have proved to be surfactant molecules that reduce surface tension and enhance cleaning ability. A significant value of detergency ability i.e. 95.94% was found in *Acacia cancina* formulation as compared to others. This can be positively correlated with the higher saponin content present in *Acacia cancina*.

Table 3: Phytochem	icals screening	of formulated	shampoos

S. No.	Herbal shampoo extracts	Total flavonoid content (Quercetin equivalents mg/g)	Total phenolic content (Gallic acid equivalents mg/g)	Total saponins content (Quillaja saponin equivalents mg/g)
1.	Aloe vera	2.19±0.06	3.71±0.07	1.56±0.05
2.	Acacia cancina	2.06±0.06	1.96±0.04	2.25±0.03
3.	Sapindus Mukorossi	1.82±0.09	1.91±0.02	1.92±0.02
4.	Phyllanthus emblica	3.33±0.04	1.92±0.02	1.64 ±0.02

Sample size, n=5

Table 4: Physicochemical	analysis of formulated shampoos

S. No.	Extracts	Dirt dispersion (Initial mo)	Dirt dispersion (After 3 mo)	Cleaning ability (Initial mo)	Cleaning ability (After 3 mo)	Detergency ability (Initial mo)	Detergency ability (After 3 mo)
1.	Aloe vera	Moderate	Moderate	86.24 %	88.00 %	75.35 %	72.69 %
2.	Acacia cancina	Moderate	Moderate	87.13 %	86.00 %	95.43 %	94.96 %
3.	Sapindus Mukorossi	Moderate	Moderate	87.86 %	94.00 %	83.82 %	79.83 %
4.	Phyllanthus emblica	Moderate	Moderate	90.89 %	96.00 %	81.34 %	78.28 %

Sample size, n=5

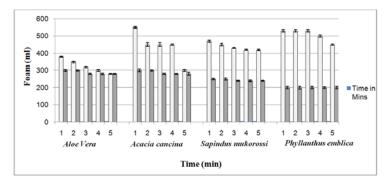


Fig. 1: Graph for the foaming ability and stability of different shampoo Values are mean±SD for three individual experiments

Foaming ability and foam stability

Foaming or lathering is an important characteristic for any formulated shampoo. It is considered to be an important parameter during commercialization of shampoos. It was observed during the studies that all the formulations produced foam above 400 ml and when tested for 5 min, it was significantly stable.

All the formulation produced rich lather formation, but a significant decrease in foaming ability was seen after 3 mo. It can be proved that the total polyphenols present in the extracts must have enhanced the foaming ability and stability of extract formulations.

CONCLUSION

Herbal shampoo with high cleansing and foaming ability was formulated using natural herbal extracts and CAPB as a conditioner. These shampoos contain high polyphenolic and flavonoid content due to which antioxidant and antibacterial activity have increased which resulted into cleaning ability with healthy hair. Thus, quality enhanced herbal shampoo was formulated with a unique aroma, colour and potential for cleaning and foaming ability.

This formulation is rich in polyphenols that have antioxidant, antimicrobial and anti-inflammatory property. A need for this age is to prepare herbal cosmetics which will prove beneficial and have lesser side effects. This will boost the use of natural medicines in the coming era.

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CONFLICT OF INTERESTS

The authors report no conflicts of interest in this work

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