

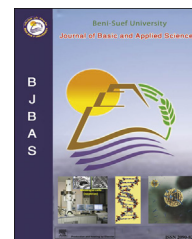
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Full Length Article

Formulation, evaluation and comparison of the herbal shampoo with the commercial shampoos[☆]

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ABSTRACT

The study aimed to formulate a pure herbal shampoo and to evaluate and compare its physicochemical properties with the marketed synthetic and herbal shampoos. The herbal shampoo was formulated by adding the extracts of *Acacia concinna*, *Sapindus mukorossi*, *Phyllanthus emblica*, *Ziziphus spina-christi* and *Citrus aurantifolia* in different proportions to a 10% aqueous gelatin solution. Small amount of methyl paraben was added as a preservative and pH was adjusted with citric acid. Several tests such as visual inspection, pH, wetting time, % of solid contents, foam volume and stability, surface tension, detergency, dirt dispersion etc, were performed to determine the physicochemical properties of both prepared and marketed shampoos. The formulated herbal shampoo was also evaluated for conditioning performance by administering a blind test to 20 student volunteers. The formulated herbal shampoo was clear and appealing. It showed good cleansing and detergency, low surface tension, small bubble size and good foam stability after 5 min. The prepared shampoo and commercial shampoos showed comparable results for % solid contents also. The score of the conditioning performance of the tress washed with herbal shampoo was found to be 3.0 out of 4, while the score of the marketed synthetic and herbal shampoo was 3.4 and 3.3 respectively. The results indicated the formulated shampoo is having excellent conditioning performance, at par with commercially available shampoo. However, further research and development is required to improve its quality and safety. Copyright 2014, Beni-Suef University. Production and hosting by Elsevier B.V. All rights reserved.

1. Introduction

Shampoos are probably the most widely used cosmetic products for cleansing hairs and scalp in our daily life (Ishi, 1997). A shampoo is basically a solution of a detergent

containing suitable additives for other benefits such as hair-conditioning enhancement, lubrication, medication etc. Now-a-days many synthetic, herbal, medicated and non medicated shampoos are available in the market but popularity of herbal shampoo among consumers is on rise because of their belief that these products being of natural origin are

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safe and free from side effects (Manikar and Jolly, 2001). Synthetic surfactants are added to shampoo primarily for the foaming and cleansing action but their regular use leads to dryness of hairs, hair loss, irritation to scalp and eyes (Potluri et al., 2013). Herbal formulations are considered as alternative to synthetic shampoo but formulating cosmetics using completely natural raw material is a difficult task (Shinde et al., 2013). There are large numbers of medicinal plants which are reported to have beneficial effects on hair and are commonly used in formulation of shampoo (Firhouse, 2009). These plant products may be used in their powdered form, crude form, purified extracts, or derivative form (Pooja et al., 2011). It is extremely difficult to prepare a herbal shampoo using a single natural material that would be milder and safer than the synthetic ones, and at the same time would compete favorably with its foaming, detergency and solid content. We, therefore, considered to formulate a pure herbal shampoo using traditionally and commonly used plant materials for hair washing in India and gulf region especially in Oman.

The pericarp of *Spindus mukorossi*, commonly known as Soapnut or reetha, fruits of *Phyllanthus emblica* commonly known as Amla, and dried pods of *Acacia concinna* (Sheekakai) have traditionally been used in Indian folklore system for centuries for washing hair (Kapoor, 2005). Reetha and Sheekakai produce rich lather when shaken with water due to their high content of saponins. They are also known to produce beneficial effects on skin and other organ systems (Khushboo et al., 2010). Amla fruit is rich in vitamin C and is employed in hair preparations as antidandruff agent, hair growth promoter and to strengthen hairs (Srivasuki, 2012). The *Ziziphus spina-christi* tree, known as Sidr in Arabic, is indigenous to the Middle East including Oman, and its leaves are traditionally used by women to wash, darken and lengthen hairs (Ali and Kadhim, 2011). It is reported to contain four saponin glycosides that help in removing excess sebum without causing adverse reactions (Mahran et al., 1996). Saponins also exhibit antibacterial and antifungal activities that make them important ingredients of cosmetic applications (Chen et al., 2010).

This study was designed to formulate a herbal shampoo and to evaluate and compare its physicochemical properties with the marketed synthetic and herbal shampoo in search of a safe and effective cosmetic product.

2. Materials and methods

2.1. Sample collection

All plant materials except *Ziziphus spina-christi* were obtained from Okhla market, New Delhi, India and were identified and authenticated by a botanist of Jamia Hamdard University. *Ziziphus* leaves were collected from the garden in Sohar, Oman and were authenticated by the Pharmacognosy professor, at Oman Medical College. Two commercially available shampoos namely Dove Shampoo® (Unilever, Middle East) and Herbal Essences shampoo® (Procter and Gamble company, US) were purchased from the local super market.

2.2. Preparation of plant extracts

100 g of *Ziziphus spina-christi* leaves were washed under running water to remove foreign substances, homogenized and boiled in hot water for 4 h. The aqueous extract was filtered and concentrated to obtain semi solid mass (yield: 11% w/w). Aqueous extracts of Sheekakai and Amla were also prepared by the similar method (yield: 8.3% w/w and 8% w/w respectively). However, Reetha pericarps were extracted by cold maceration method using 70% ethyl alcohol to obtain 11.2 g of solvent free semi solid mass (yield-11.2% w/w).

2.3. Formulation of herbal shampoo

The plant extracts were mixed in different proportions to obtain a shampoo whose formula is shown in Table 1. Herbal extracts were added to 10% gelatin solution and were mixed by shaking for 20 min. Lemon juice (1 mL) and Methyl paraben were also added with stirring. Finally the pH of the solution was adjusted by adding sufficient quantity of 1% citric acid solution. Few drops of rose essential oil were also added to impart aroma to the prepared shampoo and the final volume was made to 100 mL with gelatin solution.

2.4. Evaluation of formulated and commercial shampoo

To evaluate the quality of commercial and prepared formulations, several quality control tests including visual assessment, physicochemical controls conditioning performance tests were performed (Ashok and Rakesh, 2010).

2.4.1. Physical appearance/visual inspection

The formulation prepared was evaluated for the clarity, color, odor and foam producing ability (Aghel et al., 2007).

2.4.2. Determination of pH

The pH of 10% v/v shampoo solution in distilled water was measured by using pH meter (Mi 151, Martini instruments) at room temperature (Tarun et al., 2014).

2.4.3. Determination of % of solid contents

4 grams of shampoo were placed in a previously clean, dry and weighed evaporating dish. The dish and shampoo was weighed again to confirm the exact weight of the shampoo. The liquid portion of the shampoo was evaporated by placing the evaporating dish on the hot plate. The weight and thus %

Table 1 – Composition of formulated herbal shampoo.

Material	Quantity
Reetha extract	2.5 g
Amla extract	2.5 g
Sheekakai extract	2.5 g
Sidr extract	2 g
Lemon juice	1 mL
Methyl paraben	1 mL of 0.05% solution
Gelatin solution	q.s
Citric acid	q.s
Essential oil	0.1 mL

Table 2 – Physicochemical evaluation of formulated and marketed shampoo.

	Formulated shampoo	Dove	Herbal essences	p-value
Color	Light green	White	Light brown	
Transparency	Clear	Milky opaque	Transparent	
Odor	Good	Good	Good	
pH (10% solution)	7.02 ± 0.09	6.12 ± 0.27	6.04 ± 0.36	0.0005*
% Solid contents	22.75	25	25	
Foam volume (mL)	115 ± 3	92 ± 1.73	113 ± 1	<0.001*
Foam type	Small, dense	Small, dense	Small, airy	
Surface tension (dynes/cm)	38.72 ± 1.77	31.68 ± 2.13	38.36 ± 1.14	0.004*
Wetting time (sec)	187 ± 4	141 ± 3.46	157 ± 2	<0.001*

Results are mean ± SD (n = 3); *significant difference $p < 0.05$ by Anova single factor.

of the solid contents of shampoo left after complete drying was calculated.

2.4.4. Dirt dispersion test

Two drops of shampoo were added to 10 mL of distilled water taken in a large test tube. To this solution, one drop of India ink was added and the test tube was stoppered and shaken ten times. The amount of ink in the foam was indicated by the rubric such as None, Light, Moderate or Heavy (Ali and Kadhim, 2011).

2.4.5. Surface tension measurement

The surface tension of 10% w/v shampoo in distilled water was measured using stalagmometer at room temperature (Gaud and Gupta, 2001).

2.4.6. Test to evaluate foaming ability and foam stability

Foaming ability was determined by using cylinder shake method. Briefly, 50 mL of the 1% commercial or formulated shampoo solution was placed into a 250 mL graduated cylinder; it was covered with one hand and shaken 10 times. The total volume of the foam content after 1 min of shaking was recorded.

Foam stability was evaluated by recording the foam volume after 1 min and 4 min of shake test (Klein, 2004).

2.4.7. Wetting time test

A canvas paper was cut into 1-inch diameter discs having an average weight of 0.44 g. The smooth surface of disc was placed on the surface of 1% v/v shampoo solution and the stopwatch started. The time required for the disc to begin to sink was noted down as the wetting time (Manikar and Jolly, 2000).

2.4.8. Evaluation of conditioning performance

A hair tress of an Asian woman was obtained from a local salon. It was cut into four swatches of the tresses with approximately the length of 10 cm and the weight of 5 g. A swatch without washing served as the control. Other three tresses were washed with the commercial and formulated shampoos in an identical manner. For each cycle, each tress was shaken with the mixture of 10 g of a sample and 15 g of water in a conical flask for 2 min and then rinsed with 50 mL water. Afterward, each tress was left for air drying at room temperature. The tresses were washed for maximum ten cycles. The conditioning performance of the shampoos i.e.

smoothness and softness, was evaluated by a blind touch test, administered to twenty randomly selected student volunteers (Boonme et al., 2011). All the students were blind folded and asked to touch and rate the four tresses for conditioning performance from score 1 to 4 (1 = poor; 2 = satisfactory; 3 = good; 4 = excellent).

3. Statistical analysis

Data were analyzed using SPSS v.19. All tests were performed in triplicate and data are expressed as Mean ± standard deviation. ANOVA single factor was used for determining significance. P values <0.05 were considered as significant.

4. Results and discussion

4.1. Formulation of herbal shampoo

A pure herbal shampoo was formulated by mixing aqueous/alcoholic extracts of *Ziziphus*, Soapnut, and Sheekakai in definite amount as shown in Table 1. These plant materials contain phytochemicals such as saponins which are natural surfactants possessing good detergency and foaming properties. *P. emblica* and Sidr extracts were added as conditioning agent. A good shampoo must have adequate viscosity to facilitate removal from the bottle but must not drip down from the hair during use. A variety of natural materials is available for use as viscosity builders. We used 10% gelatin solution for this purpose as it shows pseudo-plastic behavior and forms clear solutions. Citric acid was added to adjust the pH to the desired level. Lemon juice (1 mL) was also added as natural antioxidant, chelating agent and antidandruff agent to maintain the acidic pH of formulation. Shampoo was further preserved by the addition of little amount of methyl paraben. Final formula of the prepared shampoo is presented in Table 1.

4.2. Evaluation of shampoos

Comparative effectiveness of the formulated herbal and commercial shampoo were evaluated by performing some simple physicochemical tests, results of which are discussed below.

4.2.1. Physical appearance/visual inspection

A shampoo like any other cosmetic preparation should have good appealing physical appearance. The formulated and marketed shampoos were evaluated for physical characteristics such as color, odor and transparency (Table 2). Our prepared shampoo was transparent, light green and had good odor. No significant difference was observed in terms of odor, transparency and foaming characteristics between commercial and formulated shampoo except for color.

4.2.2. pH

Most shampoos are formulated as either neutral or slightly alkaline to minimize the damage to hair. The pH of shampoo also helps in minimizing irritation to the eyes, enhances the qualities of hair and maintain the ecological balance of the scalp (Baran and Maibah, 1998). The pH of tested commercial shampoos was found within the preferred range (between 7 and 5) (Tarun et al., 2014) and are presented in Table 2. The acid balanced values were observed with commercial shampoos (Dove: 6.12, Herbal Essences: 6.04) but the pH of formulated shampoo was found to be nearly neutral (7.02).

4.2.3. % of solid contents

Good shampoos usually have 20%–30% solid content as it is easy to be applied and rinse out from the hair. If it doesn't have enough solid it will be too watery and wash away quickly, similarly too many solids will be hard to work into the hair or too hard to wash out. The percent solid contents of all the tested shampoo was found within the range of 22–25% and are expected to wash out easily (Table 2).

4.2.4. Dirt dispersion

Dirt dispersion is an important criterion for evaluation of cleansing action of shampoo. Shampoos that cause the ink to concentrate in the foam are considered of poor quality because ink or dirt that stays in foam is difficult to rinse away and gets re-deposited on the hair (Ali and Kadhim, 2011). Therefore, the dirt should stay in the water portion for achieving better cleansing action. All shampoo concentrated the ink in the water portion, ensuring their satisfactory cleaning ability and actual effectiveness.

4.2.5. Surface tension

The term indicates the amount of surfactant present in shampoo to reduce the surface tension. Lesser the surface tension stronger is the cleaning ability of the shampoo. A shampoo is considered of good quality if it decreases the surface tension of pure water from 72.28 dyn/cm to about 40 dyn/cm (Ilton et al., 2007). All the tested shampoo showed similar reduction in surface tension ranging from 31.68 to 38.72 dyn/cm. The reduction in surface tension is an indication of their good detergent action. The formulated shampoo reduced the surface tension to 38.72 dyn/cm which is comparable to Herbal Essences® (38.36 dyn/cm). However, among all the shampoo, Dove® (31.68 dyn/cm) has the lowest surface tension indicating that it has the strongest cleaning ability. The commercial synthetic or semi-herbal shampoos may contain excessive detergents, which can strip the hair of up to 80% of the oil and thus damage the hair. Using a mild detergent in our shampoo, we have ensured that this does not happen.

Table 3 – The mean score of the student volunteers opinion on the conditioning performance of the tresses after treatment with shampoos (n = 20).

Score	Formulated shampoo	Dove shampoo	Herbal essences shampoo	No washing
1	1	0	0	18
2	3	1	1	2
3	11	10	12	0
4	5	9	7	0
Average	3	3.4	3.3	1.1

Score 4 = excellent; score 3 = good; score 2 = fair and score 1 = poor.

4.2.6. Foaming ability and foaming stability

Foaming or lathering is very important to the consumer and therefore, it is considered as an important parameter in evaluation of shampoo. Herbal Essences and formulated shampoo produced the foam volume above 100 mL (115, 113 mL respectively) while dove shampoo generated a foam volume of 92 mL. The foams generated by formulated shampoo were small, compact, uniform, denser and stable similar to commercial samples. All tested shampoo had the same foam volume for 5 min showing that their foam has good stability. The higher foaming property of formulated shampoo may be due to the combination of soap nut, Sheekakai and Ziziphus (Sarath et al., 2013).

4.2.7. Wetting time

The wetting ability of a surfactant is dependant on its concentration and is commonly used to test its efficacy. The canvas disc method is quick, efficient and reliable test to evaluate the wetting ability of a shampoo (Manikar and Jolly, 2000). The wetting time of three shampoo was found in the order 141 < 157 < 187 s for dove, herbal essences and formulated shampoo respectively. It can be concluded that dove contains the maximum concentration of detergents because it had the least wetting time by contrast our formulated shampoo exhibited maximum wetting time so, it contains minimum concentration of detergents.

4.2.8. Conditioning performance

Conditioning performance of three shampoos based on the mean scores of student referees is presented in Table 3. Majority of the students rated that the tress washed with dove provided the best conditioning performance and as expected the control tress (without washing) got the minimum score (1.1). The score of the conditioning performance of the tresses washed with formulated shampoo was found to 3.0 out of 4 and was comparable with the scores of marketed shampoos. The results clearly indicated that the formulated shampoo is having good conditioning performance level.

5. Conclusion

The aim of this study was to formulate a completely herbal shampoo which is at par with the synthetic shampoo available in the market. We formulated a herbal shampoo by using plant extracts which are commonly used traditionally and

lauded for their hair cleansing actions across Asia. All the ingredients used to formulate shampoo are safer than silicones and polyquaterniums synthetic conditioning agents and vis a vis can greatly reduce the hair or protein loss during combing. Instead of using cationic conditioners we have used Sheekakai, Amla, Ziziphus and other plant extracts to provide the conditioning effects.

Several tests were performed to evaluate and compare the physicochemical properties of both prepared and marketed shampoos. Our prepared shampoo showed comparable result with that of marketed shampoo for quality control tests but further research and development is required to improve its over all quality.

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