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.

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 (Firthouse, 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 organs 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.

<table>
<thead>
<tr>
<th>Material</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reetha extract</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Amla extract</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Sheekakai extract</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Sidr extract</td>
<td>2 g</td>
</tr>
<tr>
<td>Lemon juice</td>
<td>1 mL</td>
</tr>
<tr>
<td>Methyl paraben</td>
<td>1 mL of 0.05% solution</td>
</tr>
<tr>
<td>Gelatin solution</td>
<td>q.s</td>
</tr>
<tr>
<td>Citric acid</td>
<td>q.s</td>
</tr>
<tr>
<td>Essential oil</td>
<td>0.1 mL</td>
</tr>
</tbody>
</table>
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 pseduoplastic 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.

### Table 2 – Physicochemical evaluation of formulated and marketed shampoo.

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

Results are mean ± SD (n = 3); *significant difference p < 0.05) by Anova single factor.
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 character-
istics 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 commer-
cial 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 sham-
poos (Dove: 6.12, Herbal Essences: 6.04) but the pH of formu-
lated 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 wetary 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 sur-
fase tension of pure water from 72.28 dyn/cm to about 40 dyn/
㎝ (Ilton et al., 2007). All the tested shampoo showed similar
reduction in surface tension ranging from 31.68 to 38.72 dyn/
㎝. 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>
<thead>
<tr>
<th>Score</th>
<th>Formulated shampoo</th>
<th>Dove shampoo</th>
<th>Herbal essences shampoo</th>
<th>No washing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>18</td>
</tr>
<tr>
<td>2</td>
<td>3</td>
<td>1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>11</td>
<td>10</td>
<td>12</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
<td>9</td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Average</td>
<td>3</td>
<td>3.4</td>
<td>3.3</td>
<td>1.1</td>
</tr>
</tbody>
</table>

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, Shee-
kakai and Ziziphus (Sarath et al., 2013).

4.2.7. Wetting time
The wetting ability of a surfactant is dependant on its con-
centration 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 formu-
lated 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. Ma-
jority 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 avail-
able 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 it’s overall quality.

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References
