Formulation and evaluation of Pomegranate based herbal shampoo

Tanya Malpani, Manali Jeithliya, Nanadini Pal and Payal Puri

Abstract
The aim of the present study was to formulate and evaluate herbal shampoo containing natural ingredients with an emphasis on safety and efficacy. It clears dirt, dandruff, promotes hair growth, lustre, strengthens and darkens the hair. The shampoo was prepared by taking the extracts of Pomegranate (active ingredient), Curry Leaves, Ginger, Aloe vera, and Reetha in different proportions. Several physiochemical tests were performed for visual assessment, wetting time, pH, assurance of solid contents, surface tension, detergency, dirt dispersion, conditioning performance, foam stability. The formulated herbal shampoo is black in color with demonstrable good froth stability, detergency, good cleansing, low surface tension, optimum pH and conditioning activity. Dirt dispersion of herbal shampoo is light along with 46 ml foam height. All these are the ideal characters for good quality of the herbal shampoo to be used in daily life. However, further scientific investigation is required for validation of its overall quality.

Keywords: Herbal shampoo, efficacy, pomegranate, stability, pH

Introduction
Shampoo is a hair care product, typically in the form of a viscous liquid that is used for cleansing hair. The goal of using shampoo is to remove dirt that is build up on the hair, provide nourishment and give healthy look to the hair without stripping out so much sebum from it [1]. The shampoo sector is probably the largest market for sale amongst the hair care products since shampoos are one of the cosmetic products used in daily life. Many synthetic shampoos are present in the current market both medicated and non-medicated; however, herbal shampoo are nowadays mostly popularized due to their natural origin, safety, increasing consumer demand, low cost and negligible side effects [2,3]. Herbal shampoo is a cosmetic preparation which uses herbs from plants and it is meant for washing of hair and scalp just like a regular shampoo [4, 5]. Herbal formulations are considered as alternative to synthetic shampoo but formulating cosmetics using completely natural raw material is challenging task [6, 7]. 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 well with its foaming, detergency and solid content. The selection of active ingredients for hair care is based on the ability of the ingredient to prevent skin damage as well as to improve the quality of skin by cleansing, nourishing and protecting the skin [8]. We therefore made an attempt to develop a basic protocol for herbal shampoo formulation for effective hair care.

In the present study, herbal shampoo was formulated containing suitable ingredients such as Pomegranate (Punica granatum), Ginger (Zingiber officinale), Curry Leaves (Murraya koenigii), Reetha (Sapindus mukorossi), Aloe vera (Aloe barbadensis) etc. in different proportions and evaluated for its physicochemical properties. The uniqueness of this herbal shampoo formulation is due to its active ingredient, pomegranate peel. Pomegranate peel feeds the hair from the roots, removes the fatty layer above the follicles that hinders hair growth and completely eliminates inflammation and prevents skin alopecia [9]. Its natural UV ray blocking property prevents aging in hair and makes hair shiny and soft [10]. The functional properties of the ingredients are as follows:

Function of ingredients
1) Curry leaves: It treats damaged hairs by scalp restoration process and makes them stronger. Curry leaves are rich source of beta-carotene and proteins, which reduce hair loss and increase hair growth. They also contain amino acids and antioxidants which strengthen the hair follicles and moisturize the scalp. It also helps to remove dead hair follicles, which can be reason behind its anti-dandruff property. Curry leaves are rich
source of Vitamin B, which help to restore hair color by nourishing and strengthening the roots \[11\].

2) **Pomegranate:** The polyphenols in Pomegranate peel powder combat hair loss, prevent dandruff and darken hair color. It strengthens hair follicles by stimulating circulation and improves blood flow to the scalp thereby stimulating healthy hair growth.

3) **Ginger:** Ginger has essential fatty acids, which prevent the hair from thinning. Ginger also cures dandruff since it a natural antiseptic that controls scalp problems. It also gives hair natural shine. Ginger’s active ingredients, gingerol, relax blood vessels and thereby improve the circulation of blood. This allows for the improved nourishment of hair follicles, ensuring faster hair growth \[12\].

4) **Reetha (Soap nut):** It is used as a foaming agent has been used for hair cleansing property due to high saponin content. It keeps scalp gentle & removes any microorganisms responsible for infection. It is also helpful for removal of dandruff \[11\].

5) **Aloe vera:** It helps in thickening of hair. It also helps to nourish hair \[11\].

**Function of other chemicals**
1) **Glycerine:** It helps in moisturizing of hair.
2) **Sodium Chloride:** It is used as a thickener.
3) **Sodium lauryl sulphate (SLS):** It is used as a surfactant.
4) **Lemon juice:** It is used as a preservative.
5) **Guar gum:** It is used to increase viscosity.
6) **Lavender oil:** It is used for fragrance.
7) **Activated charcoal:** It is used to impart black color.

**Materials and methods**

**Collection of plants:** The parts of plants like Pomegranate (peel), Reetha (fruit), Ginger (root) and Guar gum were collected from the local market. Curry patta (leaves) and Aloe vera (leaves) were obtained from nursery locally. These were washed under running water to remove contaminants. They are dried in sunlight (Figure 1), converted into coarse powders and sieved using 60meshes. The extracts were prepared by decoction method \[13\]. And the prepared extracts were stored in well-closed containers.

**Figure 1:** Promogranate peel and Curry leaves collected and dried

**Preparation of herbal extract:** 5g of Curry patta powder, 5g of Ginger water, 10g of Aloe vera gel, 20g of Reetha and 5g of Pomegranate peel powder (Table 1) were mixed with 100 ml water in a stainless steel vessel. The mixture was kept for boiling until the water reduced to one quarter. It was then filtered. The clear extract obtained was used as herbal extract (Figure-2).

**Table 1:** Ingredients of herbal extract

<table>
<thead>
<tr>
<th>Plant</th>
<th>Parts</th>
<th>Quantity for 100g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Curry Patta</td>
<td>Leaves</td>
<td>5%</td>
</tr>
<tr>
<td>Ginger</td>
<td>Root</td>
<td>5%</td>
</tr>
<tr>
<td>Alovera</td>
<td>Leaves</td>
<td>10%</td>
</tr>
<tr>
<td>Pomegranate</td>
<td>Peel</td>
<td>5%</td>
</tr>
<tr>
<td>Reetha</td>
<td>Fruit</td>
<td>20%</td>
</tr>
</tbody>
</table>

**Figure 2:** The dried plant materials were powdered for preparation of herbal extract

**Formulation of herbal shampoo:** Formulation of the herbal shampoo was done as per the formula given in Table 2. To increase the thickness of formulation, SLS (7.5%) solution was prepared using 0.1 M NaCl. Twenty ml of the herbal extract was added to 20 ml SLS solution with 20 ml NaCl solution and mixed by shaking gently. The final volume was made to 100 ml by adding 10 ml guar gum extract, 2 ml of glycerine and 25 ml of water. To improve aroma in the formulation, sufficient quantity (q.s.) of essential oil (lavender oil) was added. The shampoo also included one capsule of Vitamin E for conditioning, activated charcoal for color and 2 ml of lemon juice as preservative.

**Table 2:** Composition of herbal shampoo

<table>
<thead>
<tr>
<th></th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Herbal extract</td>
<td>20 ml</td>
</tr>
<tr>
<td>SLS (7.5%)</td>
<td>20 ml</td>
</tr>
<tr>
<td>0.1 M NaCl</td>
<td>20 ml</td>
</tr>
<tr>
<td>Guar gum</td>
<td>10 ml</td>
</tr>
<tr>
<td>Glycerine</td>
<td>2 ml</td>
</tr>
<tr>
<td>Vitamin E capsule</td>
<td>1</td>
</tr>
<tr>
<td>Lemon juice</td>
<td>2 ml</td>
</tr>
<tr>
<td>Essential oil (lavender)</td>
<td>q.s.</td>
</tr>
<tr>
<td>Activated charcoal</td>
<td>q.s.</td>
</tr>
<tr>
<td>Water</td>
<td>25 ml</td>
</tr>
</tbody>
</table>
Evaluation of herbal shampoo

To evaluate the prepared formulations, quality control tests including visual assessment and physicochemical controls such as pH, density, viscosity, surface tension, foam volume, foam stability and wetting time were performed using standard protocols.

1. Physical appearance/visual inspection: The formulation prepared was evaluated for the clarity, color, odor and foam producing ability and fluidity.[14]

2. Determination of pH: A 10% v/v shampoo solution was constituted in distilled water and the pH of the solution was measured by using a calibrated pH meter.[15]

3. Determination of solid content percentage: A clean dry evaporating dish was weighed and 4 grams of shampoo was added to the evaporating dish. The evaporating dish with shampoo was placed on the hot plate until the liquid portion was evaporated. The weight of the solid contents present in the shampoo was calculated after drying.

4. Wetting time: Wetting time was calculated by noting the time required by the canvas paper to sink completely.[16] A canvas paper weighing 0.44 g was cut into a disc of diameter measuring 1-inch. Over the shampoo (1% v/v) surface, the canvas paper disc was kept and the time taken for the paper to sink was measured using the stopwatch.

5. Rheological evaluation: The viscosity of herbal shampoo was determined by using Ostwald’s viscometer.[17] The viscosity of the herbal shampoo was measured by counting drops of herbal shampoo from the mark to bottom.

\[ ny = nw \times \frac{dy}{dy} \times \frac{tw}{tw} \]

- \( ny \): viscosity of water
- \( nw \): viscosity of tested liquid
- \( dw \): density of water
- \( dy \): density of tested liquid
- \( tw \): timing of runoff of water
- \( ty \): timing of runoff of tested liquid

6. Dirt dispersion: Two drops of herbal shampoo were added in a wide mouthed falcon tube containing 10ml of distilled water. 1 drop of India ink was added, the falcon tube was covered and shaken for ten times. The amount of ink in the foam was estimated as None, Light, Moderate, or Heavy.[18]

7. Cleansing action: The cleansing property of the herbal shampoo was evaluated by the application of the shampoo on hair that has not been washed for seven days. The shampoo was used to wash the hair of human subject that had applied oil 4-5 hours before washing. The performance of the shampoo was assessed on its ability to remove oily dirt from scalp.

8. Surface tension measurement: Measurement was carried out with herbal shampoo through stalagometer (Fig.3). The principle is to measure the weight of the drops of herbal shampoo falling from a capillary glass tube, and thereby can determine the surface tension of the fluid. We can determine the weight of the falling drops by counting them. From it we can determine the surface tension as shown below [19].

\[ ST = \frac{nl}{nw} \times \frac{dl}{dw} \times tw \]

- \( nl \): no. of drops of liquid
- \( nw \): no. of drops of water
- \( dl \): density of liquid
- \( dw \): density of water
- \( tw \): 71.2 dyne/cm

9. Foaming ability & foam stability: Cylinder shake method was used for determining foaming ability. 50ml of the 1% herbal shampoo solution was put into a 250ml graduated cylinder & the cylinder was covered with hands and shaken for 10 minutes. The total volume of the foam content after 1 minute shaking was recorded. Immediately after shaking the volume of foam at 1 minute intervals for 10 minutes were recorded.[20]. The foam volume remains same throughout the period of about 5 min showing that the generated foam by the shampoo has good stability and the prepared shampoo exhibits higher foam property which may be due to the presence of soapnut.

10. Stability Study: The stability of the formulation was studied for a period of four weeks by keeping at temperature of 25-30°C.

11. Skin Irritation Test: Prepared herbal shampoo was applied on skin for 5 minutes after that was washed and tested for irritation or inflammation to the skin.

12. Conditioning attributes: The conditioning effect of the shampoo on the hair was evaluated after the hair had been washed with it. Conditioning properties include all desirable benefits imparted to the hair such as increased mass to the hair, improved lusture, softness and silkiness.[21]

13. Microbial examination: 100 microlitre of shampoo was mixed with melted Mueller Hinton agar and poured to sterile petridishes under aseptic conditions. The plates were incubated at 37°C for 24 hours and observed for microbial growth.[22]

14. Anti-microbial activity: This test was carried out to determine the susceptibility or resistance of organisms to formulation ingredients according to the method described by Cheesbrough.[23] The Gram positive (Bacillus) and Gram negative (E.coli) test organisms were subcultured on nutrient broth and incubated at 37°C till desired turbidity. The developed culture was streaked on the surface of Mueller Hinton agar on which four wells were punched with sterile cork borer. 25, 50, 100 and 150 ul shampoo were filled in these wells in increasing order. The plates were incubated at 37°C for 24 hrs, and zone of inhibition around the wells were measured using a ruler.
**Results and discussion**

**Physical appearance / Visual inspection**
The formulated herbal shampoo as shown in Figure-4 is black shining in color. It has a good odor given by the fragrance in the ingredients and also a good foam producing ability.

**pH**
The pH of formulated shampoo was 6, falling within the ideal pH range for shampoo which is between 5 and 7.8. The formulated shampoo is acid balanced which is near to the skin pH. The pH of shampoo is important for improving and enhancing the qualities of hair, minimizing irritation to the eyes and stabilizing the ecological balance of the scalp. Mild acidity prevents swelling and promotes tightening of the scales, thereby inducing shine.

**Viscosity**
The viscosity of shampoo plays an important role in determining its shelf life stability, the ease of flow on removal from packing and spreading on application to hair and product consistency in the package. The viscosity of formulated shampoo was found to be 50 millipoise which was good enough for its applicability.

**Percentage of solid 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 was found to be 3% and it is suggestive that it can be washed out easily.

**Dirt dispersion**
Shampoo that causes 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. The estimated amount of ink in foam was light and so results indicate that prepared formulation is satisfactory (Figure-5).

**Cleansing action**
The cleansing action was tested on human hairs that have applied oil and not been washed for seven days. The results of detergency studies showed that the formulation has significant cleansing ability as it was able to remove both dirt and oil from hairs (Figure-6). The softness and silkiness of the hairs after hair wash attributes to the conditioning property of the herbal shampoo.

**Surface tension**
Surface tension reduction is one of the mechanisms implicated in detergency. The reduction in surface tension of water (72.8 dynes/cm) by the herbal shampoos is an indication of their good detergent action. The surface tension value of formulated shampoo was found to be 200dyne/cm³.

**Foaming ability and foam stability**
Although foam generation has little to do with the cleansing ability of shampoos, it is of importance to the consumer. The final formulation produced stable foams; (Figure-7) there was little bit change in foam volume.

**Wetting time**
The wetting ability of a surfactant is dependent on its concentration and is commonly used to test its efficacy. The canvas disc method is a quick, efficient and reliable test to evaluate the wetting ability of a shampoo. The wetting time of herbal shampoo was found to be 12 sec which is good.

**Skin irritation test**
The skin irritation tests revealed that the herbal shampoo shows no harmful effect on the skin. This is due to the absence of harmful synthetic ingredients. Most of the synthetic chemicals produce inflammation and
causes irritation to the skin. But in this formulation, almost all ingredients are obtained naturally. So it does not produce any harmful effect on the skin.

Stability study
Stability of formulations during the storage period indicated that they are chemically and physically stable. The formulated polyherbal shampoo is chemically and physically stable at standard room temperature of 25-30°C. The results indicate that it possesses good stability within the 4 weeks of stability study.

Microbiological examination
The microbiological count of the formulated shampoo was quite low (90 CFU/ml) so it is safe to use. According to Jordanian standards, shampoo formulation should not contain more than $10^2$ CFU/ml [24].

Anti-microbial activity
The result for anti-microbial activity revealed that the formulated shampoo was effective against Gram negative bacteria at all volume but with less inhibition (Figure 8). The shampoo showed no activity against Gram positive bacteria. The results were determined by appearance of circular partial zone of inhibition on agar plate around punched wells. The diameter of zone of inhibition ranged from 0.1-0.35 cm for 25-150ul volume of shampoo. The lesser inhibition may be due to high nutritive quality of pomegranate peel used. The inhibitory activity can be increased by incorporation of Neem leaves extract in the formulation.

Fig 8: Antimicrobial activity against Gram negative acteria.

Results for evaluation of formulated protein based herbal shampoo are shown in Table 3.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Parameters</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>pH</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>Percentage of solid contents</td>
<td>3%</td>
</tr>
<tr>
<td>3</td>
<td>Wetting time</td>
<td>12 sec</td>
</tr>
<tr>
<td>4</td>
<td>Viscosity</td>
<td>50 millipoise</td>
</tr>
<tr>
<td>5</td>
<td>Dirt dispersion</td>
<td>Light</td>
</tr>
<tr>
<td>6</td>
<td>Cleansing action</td>
<td>Good</td>
</tr>
<tr>
<td>7</td>
<td>Surface tension</td>
<td>200 dyne/cm²</td>
</tr>
<tr>
<td>8</td>
<td>Foam ability and stability</td>
<td>46 ml after 1 min and 41 ml after 10 min</td>
</tr>
<tr>
<td>9</td>
<td>Skin irritation</td>
<td>Nil</td>
</tr>
<tr>
<td>10</td>
<td>Microbiological count</td>
<td>90 CFU/ml</td>
</tr>
<tr>
<td>11</td>
<td>Antimicrobial activity</td>
<td>Zone of inhibition against Gram negative bacteria.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Vol of sample Zone diameter</td>
</tr>
<tr>
<td></td>
<td>25 ul</td>
<td>0.1 cm</td>
</tr>
<tr>
<td></td>
<td>50 ul</td>
<td>0.15 cm</td>
</tr>
<tr>
<td></td>
<td>100 ul</td>
<td>0.2 cm</td>
</tr>
<tr>
<td></td>
<td>150ul</td>
<td>0.35 cm</td>
</tr>
</tbody>
</table>

Conclusion
The objective of the study was to develop a stable and functionally effective herbal shampoo by excluding synthetic chemicals, which are normally incorporated in such formulations to larger extent. Synthetic hair shampoo is known to damage the hair cuticle leaving it brittle, dull and dry [13]. Although the formulated shampoo contains synthetic chemical as SLS (7.5%) but its percentage is too small as compared to synthetic shampoo (10-40%) available in the market [23]. The evaluation study on our shampoo showed good cleaning action, better foaming capacity, and quick wetting time. We have used Aloe-vera gel to provide the conditioning effects. The formulated shampoo was not only safer than the chemical conditioning agents, but also greatly reduces the protein loss during combing. The pH of the shampoo was 6 which can retain the acidic mantle of scalp. We have used lemon juice for preservation and therefore have avoided the risk posed by chemical preservatives. It is concluded that the formulated shampoo was safe and effective to use as an alternative to its synthetic counterpart. The investigation of herbal shampoo formulation was carried out for its standardization. Meanwhile, there is need to do more clinical research in this case. Herbal shampoos are generally compatible, very effective in terms of ease of manufacture and also economical. These have fewer side effects; instead provide the hair with nutrients and other useful minerals. So, in future herbal hair cosmetic is promising approach to herbal cosmetics industry.

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Conflict of interest statement
Authors have none to declare.

References