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# FORMULATION AND EVALUATION OF HERBAL SHAMPOO CONTAINING OLIVE LEAVES EXTRACT

***Hiba Yateem, Michel Hanania and Nida' Mosleh**

Department of Chemistry, Bethlehem University, Bethlehem- Palestine

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**ABSTRACT**

The study aimed at formulating a herbal shampoo containing olive leaves extract and evaluating its physiochemical properties. Olive leaves extract in shampoo is commercially available in Palestine, but because the R&D departments do not get sufficient attention neither in the private nor in the public sector, most of those products are a reproduction of what has been produced in developed countries. Moreover, there are still few data available on their stability in literature. The herbal shampoo was formulated by incorporating the ethanolic extract of olive leaves standardized for Oleuropein, which has antioxidant, anti-inflammatory and hair protectant properties. Several tests such as visual inspection, pH, percentage of the active ingredient and foam ability were conducted. Stability studies were also performed to determine the physiochemical properties of the formulated herbal shampoo. Three formulas (F1, F2 and F3) containing the same concentration of olive leave extract (1.0% w/w) were prepared. All ingredients used to formulate the shampoo were found to be safe and the physiochemical evaluation showed ideal results. Stability studies showed a stable homogenous appearance during six months of storage at different temperatures (4-8 $^{o}$C, 40 $^{o}$C and at ambient temperature). However, formula 3 gave optimum stability, especially the stability of olive leaves extract.



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## INTRODUCTION

Herbal shampoo is a type of cosmetic preparation that uses herbs from plants as an alternative to the synthetic shampoo available in the market. The herbal shampoo is important, as people nowadays prefer herbal products than chemical ones for they proved to enhance health. The awareness and need for cosmetics with herbs are on the rise, primarily because it is believed that these products are safe and free from side effects (Arora *et al*., 2011). Olive leaves has gained interest due to the numerous benefits for health, which is mainly attributed to Oleuropein, which can constitute up to 6%-9% of dry matter in leaves, as well as related derivatives (Goulas *et al.,* 2010 and Papoti *et al*., 2009). Oleuropeinhas efficacy in the management of various complex diseases including diabetes, cardiovascular disorder, viral and microbial infection as well as antioxidant properties. Olive leaves are believed to have several beneficial potentials in hair care and in medical field (Syed Hares, 2010).

***Corresponding author:*** **Hiba Yateem,**
Department of Chemistry, Bethlehem University, Bethlehem-Palestine

According to the popular belief, olive leaves helps them in various ways when used traditionally. Olive leaves are traditionally used for hair care and many people experience a noticeable improvement in just few weeks. The use of olive leaves extract in shampoo is already available commercially in the Palestinian market. But because of the R&D activities do not receive sufficient attention neither in the private nor in the public sector in Palestine especially in cosmetics, most of those products are imitated and still there are few data available on their stability in the literature. In this study, we tried to formulate a herbal shampoo containing ethanolic extract of olive leaves. The formulated shampoos were also evaluated by using standard methods.

## MATERIALS AND METHODS

All ingredients used in the formulas were purchased from BASF, Germany, except for Oleuropein (40%) which was obtained from Chengdu Biopurity Phytochemicals Ltd. China. Chromatographic grade-double distilled water; HPLC grade acetonitrile (Merck), potassium dihydrogen phosphate, phosphoric acid and ethanol were obtained from Sigma-

Aldrich Company. Chromatographic separation was achieved by LC system (Waters 1525 Binary HPLC pump) coupled with Waters 2487 Dual Absorbance detector.

**Formulations**

**Formulation of Olive Leaves Extract (Family Shampoo):** Three formulas of shampoo were prepared as shown in tables (1, 2 and 3) shown below. For the preparation of the shampoos, all ingredients were mixed together according to the formulas shown in the tables, and then olive leaves extract was added with a concentration of (1% w/w).

**Table 1. Olive leaves extract pearlized shampoo: (formula 1)**

| Chemicals | Quantity |
|---|---|
| Texapon N70 (SLS) Sodium Lauryl Sulphate | 11.60% |
| Glycol stearate | 1.80% |
| Cocamide DEA | 1.0% |
| Cocamidopropylbetaine | 1.0% |
| EDTA | 0.2% |
| Preservative | 0.02% |
| Perfume | 0.1% |
| Ammonium chloride | 2.0% |
| Olive leaves extract | 1.0% |
| Water | 81.0% |

**Table 2. Olive leaves extract clear shampoo: (formula 2)**

| Chemicals | Quantity |
|---|---|
| CocamidopropylBetaine | 3.50% |
| Cocamide DEA | 5.50% |
| Sodium laureth ether sulphate | 7.50% |
| Sodium lauryl sulphate | 3.50% |
| Polyquaternium 7 | 1.50% |
| Perfume | 0.20% |
| Citric acid | 0.15% |
| Preservative | 0.02% |
| Olive leaves extract | 1.0% |
| Water | 79.0% |

**Table 3. Olive leaves extract clear shampoo: (formula 3)**

| Chemicals | Quantity |
|---|---|
| CocamidopropylBetaine | 3.50% |
| Cocamide DEA | 5.50% |
| Sodium laureth ether sulphate | 7.50% |
| Sodium lauryl sulphate | 3.50% |
| Polyquaternium 7 | 1.50% |
| Perfume | 0.20% |
| Citric acid | 0.15% |
| Preservative | 0.02% |
| Benzophenone-4 | 0.10% |
| Olive leaves extract | 1.0% |
| Water | 77.0% |

**Evaluation of herbal shampoos:** To evaluate the prepared formulations, quality control tests including organoleptic and physiochemical characterization such as pH, solid contents and viscosity were performed. To ensure the quality of the products, specific tests for shampoo formulations including foam volume and foam stability in addition to stability study were also carried out.

**Physical appearance/ visual inspection:** All samples were observed for physical appearance/visual inspection. The prepared formulations were evaluated in terms of their color, odor, clarity and foam-producing ability.

**Determination of pH:** The pH of 10% v/v shampoo solution in distilled water was determined using appropriate pH meter at room temperature (Tarun*et al.,* 2014).

**Rheological properties:** The viscosity of the prepared formulations was measured at room temperature using a Brookfield viscometer (R/S plus rheometer model, LV, USA). 100 ml of the tested shampoo was poured in a beaker and an appropriate spindle was immersed into it. Readings were recorded after 5 min. of rotation at a speed of 10 rpm.

**Foaming ability and foam stability:** Cylinder shake method was used to test foaming ability. 20 ml of 1% of the formulated shampoo solution was placed into 100 ml graduated cylinder, covered with one hand and was shaken 10 times. After 1 min of shaking, the total volume of the foam content was recorded. Foam stability was measured or calculated by recording the foam volume after 1 min and 4 min of shake test.

**Determination of percentage of solid contents:** Four grams of the prepared shampoo were placed in a clean dry evaporating dish. The weight of the dish and shampoo was determined. The liquid portion of the shampoo was evaporated by placing it on a hot plate. After complete drying the weight of the shampoo solid contents was determined (Badi*et al.,* 2014).

**Stability Study:** Twenty-four sets of 60-gram shampoo (1% olive leaves extract) were stored at different temperatures (ambient temperature, 4-8$^{o}$C and 40$^{o}$C) for 6 months. After each month, their stability was checked regarding Oleuropein content, appearance, pH, Color and Viscosity.

**Determination of Oleuropein in shampoos by HPLC:** For determination of Oleuropein in shampoos, the reversed phase HPLC method was used with silica-based $C_{18}$ bonded phase column (5 µm, 150 × 4.6 mm inner diameter) and a mobile phase consisting of acetonitrile/ phosphate buffer pH 3.0 (20:80 v/v) at a flow rate of 1.0 ml/minute and UV detection at 280 nm (Al-Rimawi, 2014).

## RESULTS AND DISCUSSION

**The formulation of herbal shampoo:** The herbal shampoo was prepared by using water–based blend method. Three family shampoo samples were prepared, one of them is opaque which is formula 1 (F1), while the two others (formula 2 (F2) and formula 3 (F3)) are clear. Formula 1 (F1) contains water, primary and secondary surfactants. The primary surfactant is the key component in shampoos responsible for foam and cleansing such as sodium lauryl sulphate and sodium laurethsulphate. In this work, low concentration of primary surfactant (less than 12%) was used to decrease the amount of industrial ingredients in our herbal shampoo. Low foaming co-surfactant which is cocamidopropylbetaine was used to reduce the irritant (eye burning) and drying effect of the primary surfactant. Ammonium chloride was used as a highly effective viscosity enhancer that is compatible with sulphates. Cocamide DEA was used as a foam booster, while glycol stearate was used as an opacifier. EDTA was used as chelating agent, while color and perfume gave the final product a marketable appearance. Finally, a preservative was added to avoid spoiling. Formulas 2 and 3 are clear family shampoo which contain water, primary surfactant (with concentration less than 12%), secondary surfactant, foam booster, conditioning agent (polyquaternium 7), citric acid to adjust pH and finally color, perfume and preservative.

**Table 4. Physiochemical evaluation of formulated herbal shampoo containing olive leaves extract**

| Parameter | Formula 1 | Formula 2 | Formula 3 |
|---|---|---|---|
| Color | Green | Brown | Brown |
| Transparency | Opaque | Clear | Clear |
| Odor | Good | Good | Good |
| pH (10% solution) | 5.5 | 6.8 | 6.8 |
| Foam-producing ability | Yes | Yes | Yes |
| Percentage of solid contents | 26.0% | 25.0% | 25.0% |
| Foam volume | 80 ml | 85 ml | 85 ml |
| Foam type | Small, dense and uniform | Medium, dense and uniform | Medium, dense and uniform |
| Foam stability and uniform | Good | Good | Good |
| Viscosity (mPa.s at RT) | 50,000 | 30,000 | 30,000 |

**Table 5. Effect of storage time on the physical properties, pH and viscosity of the herbal shampoo after 12 months at ambient temperature**

| | Formula 1 | | Formula 2 | | Formula 3 | |
|---|---|---|---|---|---|---|
| | Freshly prepared | 12 months | Freshly prepared | 12 months | Freshly prepared | 12 months |
| pH | 5.5 | 5.6 | 6.8 | 6.7 | 6.8 | 6.8 |
| Viscosity (mPa.s) | 50,000 | 50,000 | 30,000 | 30,000 | 30,000 | 30,000 |
| Color | Green | Green | Brown | Brown | Brown | Brown |
| Odor | Good | Good | Good | Good | Good | Good |

**Table 6. Results of stability of Oleuropein content determined by HPLC analysis**

| Parameter | F1 | F2 | F3 |
|---|---|---|---|
| T1 at ambient temperature | N.R. | 100% | 100% |
| T1 at (4-8 °C) | N.R. | 100% | 100% |
| T1 at 40 °C | N.R. | 98.8% | 100% |
| T2 at ambient temperature | N.R. | 97.2% | 99.8% |
| T2 at (4-8 °C) | N.R. | 84.7% | 99.7% |
| T2 at 40 °C | N.R. | 74.5% | 99.5% |
| T4 at ambient temperature | N.R. | 82.0% | 99.7% |
| T4 at (4-8 °C) | N.R. | 70.3% | 99.6% |
| T4 at 40 °C | N.R. | 60.4% | 99.4% |
| T6 at ambient temperature | N.R. | 65.4% | 99.0% |
| T9 at ambient temperature | N.R. | 55.4% | 98.9% |
| T12 at ambient temperature | N.R. | 44.0% | 98.7% |

T1: One month, T2: Two months, T4: Four months, T6: Six months, T9: Nine months, T12: Twelve months. N.R. = No results.

The only difference between the two formulas is the presence of Benzophenone-4 in formula 3, which acts as UV absorber. The incorporation of benzophenone-4 allows for studying the effect of this material on the stability of Oleuropein in olive leaves extract, especially when using clear packaging rather than opaque as primary packaging in the final industrial process. Olive leaves extract (40% Oleuropein) which was chosen in our formulations is the same material used by the local Palestinian cosmetics factories.

**Evaluation of Shampoo:** The formulated herbal shampoos containing olive leaves extract were evaluated for several physicochemical properties and the results are summarized in Table 4.

**Physical appearance:** As it isclearly seen in table 4, all formulations showed good characteristics with respect to appearance and foaming.

**pH:** The pH balance of products is important as it affects skin and the surface on which they are used. The pH of our formulated shampoos fall within the ideal pH range for shampoo, i.e. 5- 7.

**Percentage of solid contents:** The percentage range of solid contents of well-prepared shampoos is 20-30% (AlQuadeib *et al*., 2018). Low amount of solid will result in watery formulation which can be washed away quickly. However, if too many solids are present, it is difficult to wash out.

The solid contents of the prepared formulas were in the range of 25.0 to 26.0%. Thus, they are considered easy to wash-out shampoos.

**Foam ability and foam stability:** One of the essential parameters in evaluating a shampoo is lathering or mostly described as foaming. The herbal shampoo using olive leaves extract resulted in the formation of small-medium, dense and uniform type of foam. The foam volume remained unchanged during a 5-minute period which suggests that the produced foams have good stability.

**Viscosity:** Viscosity plays an important role in defining and controlling many attributes of the product such as shelf life stability, clarity, ease of flow, package removal, consistency and degree of spreading upon application on hair. The flow characteristics of non-Newtonian materials are usually not measured with a single data point, because their viscosity is approximately measured as compared to those of the process being modeled (Miner, 1993). As shown in table 4, the average viscosities were within limit, which indicates suitability of shampoos for applications on hair.

**Stability study:** Accelerated stability tests (Tables 5 and 6) showed that pH of the shampoos was stable and suitable for hair application (F1= 5.6, F2= 6.7 and F3= 6.8). Viscosities were stable over the study period (50,000 mPa.s for F1, 30,000 mPa.s for F2 and F3). Color and odor were stable with no observed significant changes for the three formulas. Samples

were placed in opaque plastic bottles and stored in a chamber at temperatures 18-30$^{o}$C (Sekar, 2016). Stability studies (Table 5) showed a stable homogenous appearance during a 12 month storage period and no separation of phases occurred. The Oleuropein content (Table 6) was stable in formula 3 due to the presence of Benzophenone-4 in this formula, which acted as UV absorber. The Oleuropein content decreased with storage time in formula 2 due to its heat sensitivity. However, formula 1 did not give any result probably because of the presence of pearling agent, glycol stearate, which kept Oleuropein in suspension. In table 6, the degradation profile of Oleuropein incorporated into shampoo is shown. In addition, this table provides important data for the establishment of the shelf life of this product especially that of formula 3. As a rule of thumb, one month storage at 50$^{o}$C is equivalent to 8 months stored at room temperature, whereas three month-storage at 37$^{o}$C is equivalent to one year at room temperature. Consequently, the expiry date of formula 3 shampoo can be set to 3 years, and the consumer will still have a sufficiently active concentration of product efficacy.

**Conclusion**

This study aimed at preparing stable formulas shampoo rich with olive leaves extract that contains low surfactants to reduce the risk of chemicals. Results showed that all ingredients used to formulate the shampoo were found to be safe and the physiochemical evaluation showed ideal results. Stability studies showed a stable homogenous appearance during six months of storage at different temperatures (4-8$^{o}$C, 40$^{o}$C and at ambient temperature). However, formula 3 (F3) gave optimum stability, especially the stability of olive leaves extract. Further research is required to improve its quality especially on the conditioning performance.

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