

HERBAL SHAMPOO FORMULATIONS, THEIR ASSESSMENT FOR HAIR CLEANING AND ANTIFUNGAL ACTION

SADIA ZULFIQAR

Department of Basic & Applied Chemistry, University of Central Punjab, Lahore Pakistan.

Dr. KHALIDA NASEEM

Assistant Professor, Department of Basic & Applied Chemistry, University of Central Punjab, Lahore Pakistan. Corresponding Author Email: khalidanaseem1@gmail.com, khalida.naseem@ucp.edu.pk

AMER SHAKEEL

School of Commerce & Accountancy, University of Management & Technology, Lahore Pakistan.

ZULKIFLE IKRAM

Department of Basic & Applied Chemistry, University of Central Punjab, Lahore Pakistan.

MUHAMMAD NABEEL

Department of Basic & Applied Chemistry, University of Central Punjab, Lahore Pakistan.

AYESHA SADIQA

Department of Chemistry, University of Lahore, Pakistan.

Abstract

This study aimed to formulate polyherbal shampoos by using different herbs and comparison of their properties and functioning with marketed herbal shampoo. Shampoo formulations were prepared by using the extracts of *Allium sativum*, *Punica granatum* and *Azadirachta indica*, along with others biological extracts such as *Citrus limon*, *Moringa oleifera*, *Sapindus mukorossi*, *Phyllanthus emblica*, *Acacia concinna*, *Linum usitatissimum*, *Murrayakoenigii*, *Lawsonia inermis* and *Trigonella foenum* in their varying amounts due to their anti-fungal/antidandruff activities. For this purpose, different extraction methods such as decoction, soxhlet extraction and maceration were adopted to prepare herbal extract. All the formulations were clear and appealing. Yet, best results were obtained in case of F5 formulation. F5 formulation in which high amount of different herbal extract such as garlic, neem, shikakai, hinna, linseed was used showed sharp decrease in water surface tension, efficient cleaning ability, high emulsion percentage, high wetting ability, good foaming action and foam stability as compared to the other formulation. Yet, its texture was slightly tough as compared to marketed shampoo and other formulations. It was also observed that physiochemical properties of F5 formulation were much better than commercially available tested herbal shampoo. Average foam volume observed in first five minutes of shaking the formulation was found as 177 mL that is high in case of commercial herbal shampoo with volume found as 103 mL. F5 formulation also showed high wetting time along with compatible pH value, surface tension and degeneracy power. Thus, this assessment shows some results to be even better than the commercially available herbal shampoo which depicted that the produced shampoo is a good competitor against shampoos available commercially.

Keywords: Herbs, Shampoo, Antidandruff, Physiochemical Analysis, Extracts.

1. INTRODUCTION

Human hair exhibits a great worth because they actually play an integral part in the beauty of a person. Since ancient times, people have taken care of their hair in order to cleaning, managing and beautifying their hair. They washed and cleaned them with different materials such as herbs and ashes to gain beauty [1, 2]. People were also accustomed to wash their hair using common soap. The soap, however, left a thin film on the hair which transfer the hair unhealthy-look [3]. Animal fats and plants oil have also been reported to be used for cleaning and beautifying purpose of hair [3] .

Now a days, shampoo is a product that is used for cleaning the hair from dust and oil to feel better and look beautiful [4]. It is used for the purpose of cleaning to get rid of accumulated sebum and dirt present on the scalp [5]. The added functionalities of shampoo also include lubrication, conditioning and medication [6, 7]. Yet, shampoo formulation should be medically safe for long term use. Current shampoo manufacturing companies are launching extremely diverse and specialized shampoos, one after another, at a very rapid rate. Commercial shampoos contain various ingredients that play an active role in the cleanliness and treatment of hair. However, these ingredients might result in hair dryness, dullness and even early graying of hair [3].

According to reports, shampoos have been cauterized into two types such as synthetic and herbal shampoos depending upon the ingredients used for their preparation. Synthetic shampoos are formulated by adding some chemicals to get fast results. However, they may cause damage to hair and results in hair fall, decrease hair volume and hair growth. When synthetic shampoos are used for a prolonged period of time, some components present in them may start showing their adverse effects in form of greying hair. Yet, as compared to synthetic shampoos, herbal shampoos are known widely for having no side effects even when they are used for a very long time. This aspect makes them more favorable for their wide range use. In the last few years, trends have been shifted towards the use of herbal instead of synthetic shampoos due to adverse effects of used chemicals. Natural shampoos contain only natural ingredients such as tulsi, neem, amla, reetha, chandan, shikakai *etc.* Different parts of these plants are used in the production of such shampoos, by producing their herbal extracts using various methods like decoction, Soxhlet extraction and maceration. Various other herbal products can also be added into the shampoos to enhance their required useful properties such as anti-dandruff, anti-fungal, anti-bacterial and anti-microbial properties.

Many studies have been reported about the use of neem, garlic or pomegranate peels as a potential antiseptic, anti-oxidant, antidandruff agent in shampoo formulations, separately. Yet, no one reported the use of all these contents collectively to get synergistic properties. Current formulations contain all these ingredients to get the shampoo formulation with better properties as compared to previously reported work. Here, preparation of different shampoo formulations contained different herbs extract and biological ingredients have been reported to get clean and healthy hair. Herbal extract was prepared by different methods such as decoction, soxhlet extraction and maceration.

These extracts were added in different proportions to get different formulations and their physicochemical properties were studied via different techniques.

2. MATERIALS AND METHODS

2.1 Chemicals

Methyl paraben, NaOH, guar gum, gelatin and sodium lauryl sulphate were used as such as received without any purification.

2.2 Collection and Drying of Herbs

Different plants such as *Allium sativum* (garlic), *Punica granatum* (pomegranate) and *Citrus limon* (lemon) were purchased from the local market of Lahore, Pakistan. Other ingredients like *Moringa oleifera* (suhanjna), *Sapindus mukorossi* (reetha), *Phyllanthus emblica* (amla), *Acacia concinna* (shikakai), *Linum usitatissimum* (linseed), *Murraya koenigii* (curry leaves), *Lawsonia inermis* (henna) and *Trigonella foenum* (fenugreek) were obtained in dried form from Valencia market, Lahore Pakistan.

Aloe vera, Eucalyptus and *Azadirachta indica* (neem) were sourced from a local plant nursery. A polyherbal shampoo was also sourced from Carrefour, private limited, Lahore Pakistan for comparative analysis.

Herbs were cleaned by rinsing them with running water, placed under shade in order to dry and covered with a piece of cloth so it may not come in contact with dust particles or any foreign particles [8].

2.3 Preparation of Herbal Extracts

To prepare the herbal extracts, decoction, maceration and Soxhlet apparatus method were used. Decoction and maceration methods are simple and do not require high volumes of expensive solvents.

These methods are also reliable for better extraction of biochemical natural products present in the plants.

Soxhlet method was used where the yield reported in literature, for a plant was lower using decoction and maceration methods.

Soxhlet apparatus reuses the same solvent to maximize the extraction, however, this technique is time consuming and requires high volume of solvent.

2.3.1 Decoction Method

In decoction method, dried herbs were thoroughly ground into a fine powder using a grinder (NDNational Grinder JB-222). Powdered herbs were then sieved using a 100 µm mesh US standard steel sieve.

The sieved powdered samples were taken in glass containers along with distilled water, separately. The ratio of water to powdered samples was kept as 16:1 (400 mL of distilled water for 25g of sample).

Then this mixture was heated at 100 °C until almost one-third of it was left. Then, these contents were allowed to cool and filtered.

The filtrate was simmered until a quarter of the initial sample solution was left. After that, prepared extracts were stored in refrigerator at 4°C for further analysis and use [9].

2.3.2 Soxhlet Method

In Soxhlet extraction method, 5 g of the powdered sample was placed in a porous thimble along with the ethanol used as solvent and placed in the extraction chamber of the apparatus.

The ratio of the solvent to the sample was 100:1 like 5 g of garlic powder along with 500 mL of ethanol.

The solvent temperature was maintained at 80° C for almost 6 hours. The extraction was continued until all the soluble components were obtained in the round bottom flask.

After the extraction, the solvent was evaporated using a rotary evaporator and the extract was preserved for further use [10].

2.3.3 Maceration Method

In maceration process, 20% ethanol was used as solvent. The powdered sample was added into a glass beaker having ethanol solution in it. The ratio of the sample to ethanol was kept as 1:20 (50 g of sample in 1000 mL of 20 % ethanol).

The beaker was completely covered with aluminum foil and sealed with woolen thread. The beaker was shaken periodically. The contents of this beaker were filtered after 7 days.

The filtrate was simmered at low temperature until ethanol was evaporated. After simmering, the prepared extracts were preserved for further use [11].

2.4 Formulation of Herbal Shampoo

Different herbal extracts were mixed in definite quantity in 100 mL glass beakers to obtain five different polyherbal shampoo formulations designated as F1, F2, F3, F4 and F5.

The amount of extracts used to prepare five different formulations are given in **Table 1**.

In each formulation, methyl paraben was added as preservative, few drops of 10% solution of citric acid or 10% solution of NaOH were added to maintain the pH in range for shampoo formulations.

To control the viscosity of each formulation, guar gum or gelatin solution was also added according to the need.

Sodium lauryl sulphate was added to increase the foaming potential of the formulations. Prepared formulations were tested for different physiochemical properties.

Table 1: Composition of Shampoo Formulations

Ingredients	F1 (mL)	F2 (mL)	F3 (mL)	F4 (mL)	F5 (mL)
Garlic	1.2	1.6	2	2.4	2.8
Pomegranate	16	14	12	10	8
Neem	6	8	10	12	14
Eucalyptus	14	12	10	8	6
Lemon	2.8	2.4	2	1.6	1.2
Amla	12	10	8	6	4
Shikakai	10	12	14	16	18
Henna	4	6	8	10	12
Aelo Vera	8	8	8	8	8
Linseed	6	8	10	12	14
Curry Leaves	2	2	2	2	2
Fenugreek	4	4	4	4	4
Moringa Oleifera	14	12	10	8	6
Guar Gum	q.s				
Sodium Laurayl Sulphate	q.s				
Methyl Paraben	q.s				
10% NaOH solution	q.s				

*q.s-quantum satis-as much as required

2.5 Physiochemical Analysis for Qualitative and Quantitative Study

Several analyses such as organoleptic tests were performed to study the physiochemical properties of the prepared shampoo formulations along with commercially available herbal shampoo for comparison purpose. In organoleptic test, the formulations were assessed for color, odor, taste and texture. In pH determination test, 10% v/v solution of shampoo in distilled water was prepared for each formulation and pH was determined by a pH meter (Hanna® Instruments HI 9813-5 pH/EC/TDS meter) at room temperature [12]. Solid contents in shampoo were also evaluated. For this purpose, specific amount of shampoo was placed in a pre-weighed evaporating dish. Dish containing the shampoo was weighed to confirm the exact weight of the shampoo Then, hot plate was used to evaporate all of the liquid contents from the shampoo. When all liquid content was evaporation, the formulation was again weighed. Dried weight and initial weight was used to find the percentage of solid contents in the shampoo formulation while using the equation 1.

$$\text{Solid contents (\%)} = \frac{\text{Weight of dried shampoo}}{\text{Initial weight of the shampoo}} \times 100 \quad (1)$$

In order to determine the dirt dispersion property of the formulations, few drops of shampoo were added to 10 mL of distilled water in a test tube. Blue ink was added to the test tube and shaken vigorously for 10 times. Shaking of tube caused the formation of foam as shown in Figure 1(a). The foam was analyzed for the presence of ink quantity. Evaluated ink quantity in form of foam was reported in terms of none, light, moderate and heavy appearance of ink [13].

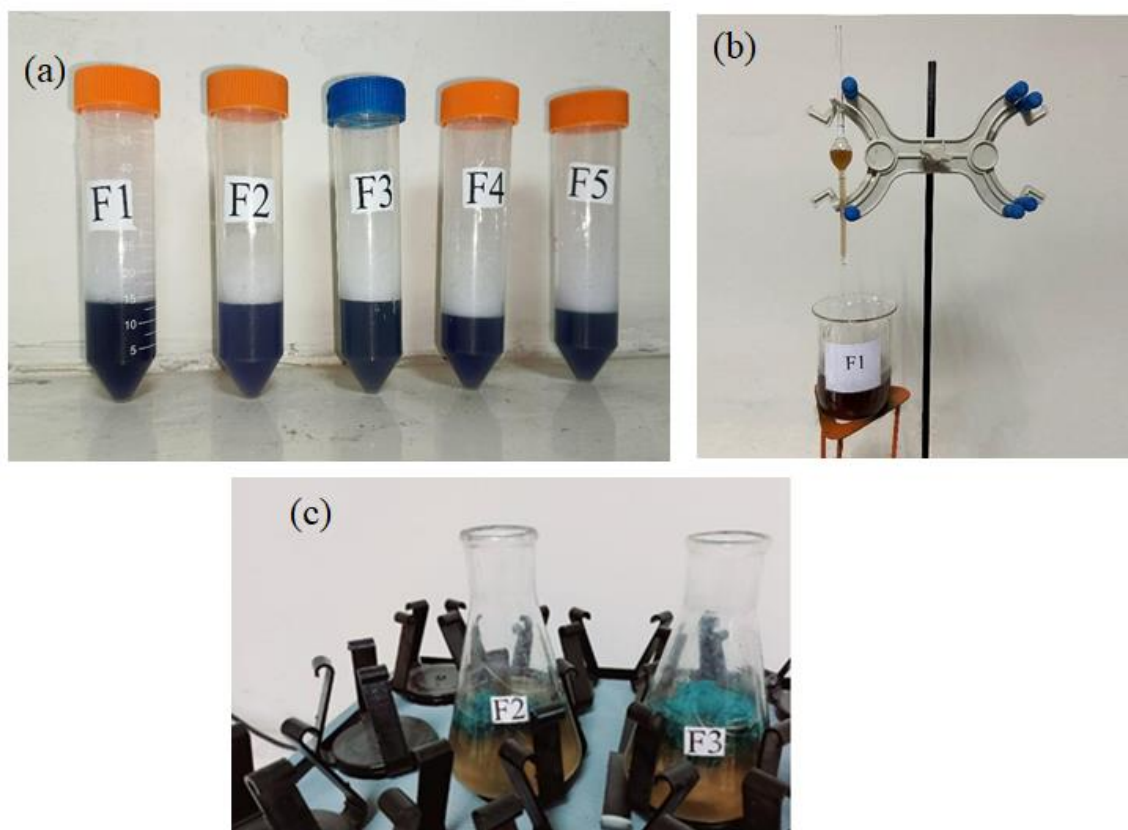


Figure 1: Analysis of Shampoo Formulations (a) Dirt Dispersion Test, (b) Assessment of Surface Tension and (c) Assessment of Cleaning Action

Value of surface tension of all prepared formulations was also measured by drop count method while using the stalagmometer. For this purpose, 10% solution of shampoo was prepared in distilled water for each formulation. Flat end of the stalagmometer was dipped in the solution and the solution was sucked up to the marked point. It was then fixed in a stand with a beaker under it as shown in Figure 1(b). The solution was then allowed to run and the number of drops were counted as it flows from high to low point. This method was repeated for distilled water and surface tension of the shampoo formulations was calculated by using the equation 2 [5].

$$R_2 = \frac{(W_3 - W_1)N_1}{(W_2 - W_1)N_2} \times R_1 \quad (2)$$

In equation 2, W_1 represents the weight of specific gravity bottle, W_2 is the weight of specific gravity bottle with distilled water. W_3 is weight of specific gravity bottle with shampoo solution. Number of drops of distilled water and shampoo are denoted by N_1 and N_2 , respectively. R_1 is the surface tension of distilled water while R_2 is the surface tension of shampoo solution.

In order to determine the cleaning action of formulations, small portion of wool yarn covered in grease was added to a 250 mL conical flask containing 1 g of shampoo in 200 mL distilled water at room temperature. Flask was thoroughly shaken for 4 minutes at the speed of 50 rpm as shown in Figure 1(c). After that, wool yarn was taken out, dried and then weighed. The detergency power was calculated by using the equation 3 [5].

$$DP = 100 \left(1 - \frac{T}{C} \right) \quad (3)$$

In equation 3, DP is the percentage of detergency power, T represents weight of sebum in the washed sample and C is the weight of sebum in the unwashed sample. In order to evaluate the foam volume and stability, cylinder shake test was performed. For this purpose, 50 mL of 1% shampoo solution for all formulations was taken in a graduated cylinder, separately. The cylinder was covered and shook 10 times then after one minute the volume of foam was recorded as shown in Figure 2(a). For the stability, the foam volume readings were taken at one minute interval [13].

A one-inch diameter disc of a canvas paper was cut in order to perform wetting time test. The weight of the disc should not exceed from 0.44 g in order to perform the test successfully. Then, distilled water along with the shampoo formulation was added to a beaker to prepare 1% v/v shampoo solution and the disc was placed onto the surface of this solution, having smooth side facing the solution surface as shown in Figure 2(b). A stopwatch was used to measure the time for the disc to completely come in contact with water, soak fully and begin sinking. This time was noted as the wetting time of the shampoo for all the formulations, separately [14].

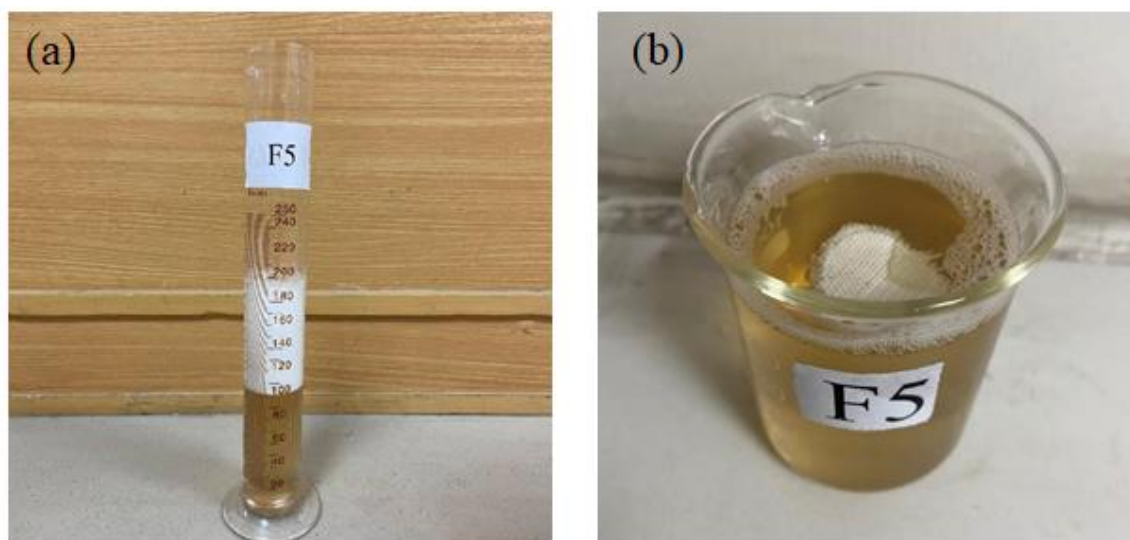


Figure 2: Assessment of (a) Foaming Stability Using Cylinder Shake Test and (b) Wetting Time Test for Shampoo Formulations

Solubility test was performed by taking 100 mL of distilled water and 2 mL of shampoo in a beaker. Shampoo formulation solution was stirred thoroughly and continuously. It was then heated to dissolve the maximum amount of shampoo. Solution was allowed to cool for 10 minutes and then the quantity of any residue of the shampoo was measured if exists any [15]. To perform the emulsion test, a test tube was taken and 1 mL of shampoo along with 1 mL of coconut oil was added in it. In another test tube, 1 mL of coconut oil was added but instead of shampoo, 1 mL distilled water was taken which acted as blank. Each test tube was shaken for almost 60 seconds and then allowed to rest for 24 hours. The height of the emulsion was noted along with the total height of the of the sample and percentage of emulsion was calculated using formula given in equation 4.

$$\text{Emulsion formation (\%)} = \frac{\text{Height of the emulsion}}{\text{Total height of the sample}} \times 100 \quad (4)$$

In order to perform stability test, each of the freshly prepared shampoo formulations were taken into test tubes separately, tested for their pH, foaming stability, surface tension and organoleptic properties and results were recorded. The shampoo formulations were stored at room temperature for 2 weeks. After 2 weeks, the test for foaming ability, pH and surface tension for each formulation was repeated. Repetitive testing assessed the stability of the shampoo.

In order to test the anti-fungal potential of the shampoo formulations, diluted shampoo formulations were added to a fungus specie grown in agar gel. First of all, a culture of *Malassezia furfur* was prepared, which was then added at random spots on an agar gel by dipping a cotton bud in the culture and rubbing it onto the agar gel. Filter paper discs, 6 mm in diameter were soaked in the shampoo formulations and placed on the agar gel. The agar gel plate was incubated for 48 hours at 25-35°C. Because of the incubation, fungal species started to grow. The shampoo formulations from the filter paper slowly diffused into the gel over time. As the fungus grew, it came incontact with the shampoo. As shampoo formulations exhibit anti-fungal properties, a zone of inhibition was observed. This zone of inhibition is the area where shampoo was present and did not allow the fungus to grow. The diameter of zone of inhibition for each formulation was measured using a scale and reported. All tests were performed in triplicate and data has been expressed as mean \pm standard deviation. Data was also analyzed statistically by using SPSS v.19.

3. RESULTS AND DISCUSSION

Mass of different plants collected for the preparation of shampoo formulations was measured before and after drying as shown in **Figure 3(a)**. It can be seen that major difference in mass of fresh and dried leaves of lemon, eucalyptus and neem was observed. Small different in mass was observed in case of pomegranate and garlic before and after drying process. Studies indicate that neem, garlic or pomegranate peels act as a potential antiseptic, anti-oxidant, antidandruff agents [16]. Use of these extract induce the synergistic properties in shampoo formulations for healthy growth of hair.

3.1 Characterization of Shampoo Formulations

A number of shampoo formulations were prepared by varying the amount of herbal extracts. Primary focus was the production of shampoos containing all three basic ingredients such as neem, garlic and pomegranate peels. Lavender was added in all the formulations according to need for odor. These formulations were tested on various grounds to explore the collective performance of all the added ingredients. Results for organoleptic test are given in **Table 2**. These results show that all the prepared shampoo formulations were dark brown in color and had a bitter taste. The odor of the shampoo was pleasant as natural perfuming agent. However, F4 and F5 gave a slightly pungent smell because of higher concentration of garlic present in these formulations. The texture of F1, F2 and F3 was smooth but the texture of F4 and F5 was slightly rough because of the use of high amount of linseed in them. Yet, the taste of all formulations was bitter. In case of reference herbal shampoo, color was greenish, texture was smooth and it had a pleasant odor, yet, the taste was found bitter. Thus, results indicate that our prepared formulation such as F1, F2 and F3 were found compatible with the herbal shampoo available in market in odor, taste and texture.

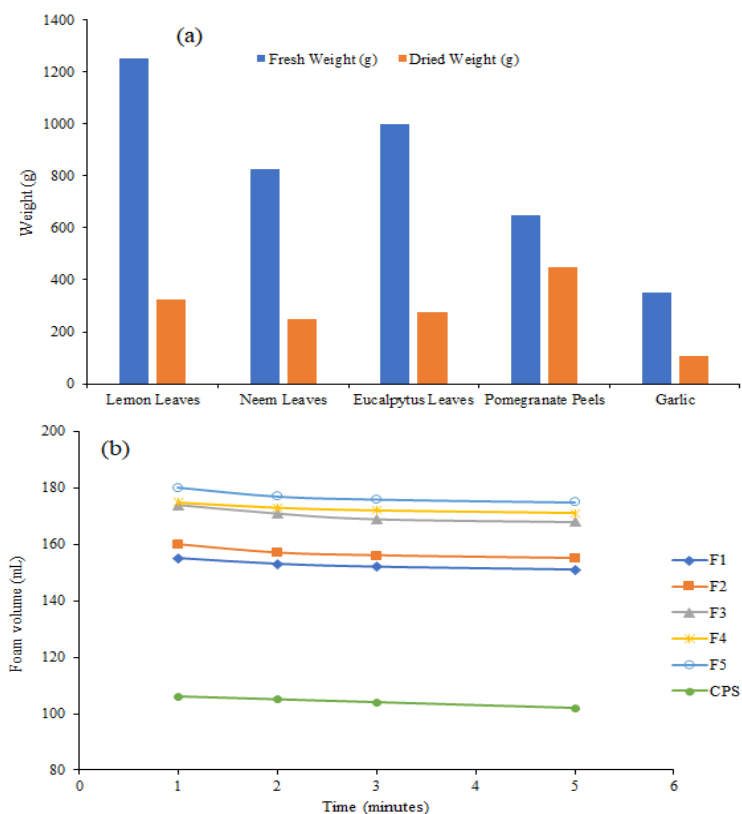


Figure 3: (a) Comparative Representation of Fresh and Dry Weights of the Plants used in Formulations, (b) Graphical Representation of Volume of Foam Produced and its Stability

Table 2: Results of Organoleptic Test, Wetting Time and Dirt Dispersion Test

Formulations	Organoleptic test				Wetting time (sec)	Amount of ink in foam
	Color	Odor	Taste	Texture		
F1	Dark Brown	Pleasant	Bitter	Smooth	230	Moderate
F2	Dark Brown	Pleasant	Bitter	Smooth	241	Light
F3	Dark Brown	Pleasant	Bitter	Smooth	220	Light
F4	Dark Brown	Slightly pungent	Bitter	Slightly rough	213	Light
F5	Dark Brown	Slightly pungent	Bitter	Relatively rough	250	Moderate
CPS	Green	Pleasant	Bitter	Smooth	84	Moderate

It has been reported that clearer the foam of a shampoo makes its quality better [1]. Dirt dispersion test was performed to check the quality of shampoo to remove unwanted particles from the hair. For this purpose, ink was added in the shampoo and test was performed. The presence of ink in the foam after conducting the test indicates that the ink would not be rinsed off and will stay in the hair. Results reported in Table 3 showed that F1 and F5 have moderate amount of ink in the foam after performing the test while F2, F3 and F4 had very light to no ink present. This indicates that the use of F2, F3 and F4 formulation on hair will keep the unwanted sebum and dirt in the water and can be easily rinsed off from the hair. Reference herbal shampoo also showed moderate amount of ink present in the foam. It illustrates that our prepared F2, F3 and F4 formulations are better than market reported herbal shampoo.

Foaming formation increases the market value and the visual appeal of a shampoo. The prepared shampoo formulations showed good foam volume and foaming stability. Reference herbal shampoo showed the initial foam volume of 106 mL, but after five minutes, the foam volume was recorded as 101 mL as given in **Table 3** which has been shown graphically in **Figure 3(b)**. It was seen that F1 displayed the lowest amount of foam formation. Yet, this value was significantly higher than that of the foam volume recorded in case of reference herbal shampoo. In this formulation, initial foam volume was noted as 155 mL and after 5 minutes, the volume was found as 151 mL. The highest foam volume was recorded in case of F5 formulation with value found as 180 mL. After 5 minutes, the foam volume of F5 was decreased to 175 mL. The reason of increase in the foam volume when moving from F1 to F5 can be due to the use of increased amount of shikakai which contains saponin contents. Saponin contents enhance the foam formation in the shampoo formulations [17]. Comparative representation of formation of foam volume in case of all formulations and herbal shampoo used as standard is shown in **Figure 3(b)**.

Table 3: Results of the Foaming Formation Ability and Foam Stability Test

Formulations	Foam volume in freshly prepared formulations (mL)				Foam volume in two weeks old formulations (mL)			
	1	2	3	5	1	2	3	5
Shaking time (min)								
F1	155	153	152	151	158	157	156	153
F2	160	157	156	155	164	162	161	159
F3	174	171	169	168	180	177	176	171
F4	175	173	172	171	183	182	180	177

F5	180	177	176	175	185	184	183	180
CPS	106	105	103	101	108	106	104	101

Table 4: pH, Solid Content, Surface Tension and Detergency Test with the Stability for Shampoo Formulations

Formulations	pH		Percentage of solid contents (%)	Surface tension (dynes/cm)		Detergency power (%)
	Freshly Prepared	Two weeks old		Freshly Prepared	Two weeks old	
	F1	5.33 ± 0.04		5.65 ± 0.02	28.00	
F2	5.59 ± 0.01	5.92 ± 0.03	25.24	30.95	31.16	24.36
F3	5.46 ± 0.04	5.87 ± 0.01	29.52	28.45	28.56	24.73
F4	5.72 ± 0.02	6.06 ± 0.04	21.51	28.08	27.91	26.66
F5	5.83 ± 0.03	6.14 ± 0.02	21.09	27.29	27.67	28.26
CPS	6.72 ± 0.01		9.50	31.51	30.54	22.02

The pH of human scalp is acidic and the diversity of microorganisms that live on the scalp survive in acidic conditions. Furthermore, alkaline shampoo formulations make hair frizzier and more prone to damage. The ideal pH range for shampoo formulation is 5.0-7.8 to overcome these problems [18]. The lowest pH observed in current study was 5.33 ± 0.04 found in case of F1 formulation while the highest pH was 5.83 ± 0.03 observed in case of F5. pH value observed in reference herbal shampoo was found as 6.72 ± 0.01 and given in Table 4. Graphical representation of different shampoo formulation as function of their pH are shown in **Figure 4(a)**. It was concluded that pH values of all formulations were found in ideal range and are proved according to the standard.

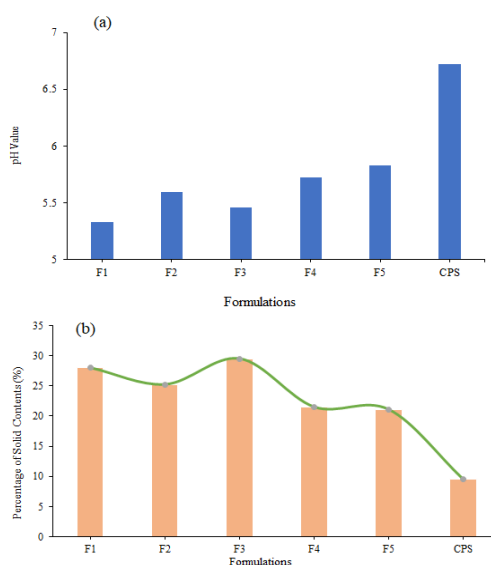


Figure 4: Representation of Different Formulations with Respect to their (a) pH Values and (b) Total Solid Contents

Present study shows that the highest solid contents reported was 29.52 % in case of F3 formulation while the lowest value was found as 21.05 % in case of F5. Reference herbal shampoo had a solid contents has 9.5%. Solid contents found in different formulations along with standard shampoo is shown in **Figure 4(b)**.

According to the standard, acceptable total solid contents found in shampoo formulations should be in range of 20-30 % [19]. The reason is that presence of high percentage of solids contents makes its removal difficult from hair during washing process [20]. Thus it was concluded that our prepared shampoo formulations were found in acceptable range with respect to the percentage of solid contents.

Another property important to make a good shampoo is its ability to low the surface tension value of water up to 40 dynes/cm from 72.8 dynes/cm [21]. More the shampoo lowering the surface tension value of water, the better is its functioning in cleaning the hair. Low surface tension of water allows it to reach deep crevices and do thorough cleaning of the scalp.

High decrease in surface tension of water was observed in case of F5 formulation while lowest decrease was observed in case of F2 formulation with the values found as 27.291 and 30.955 dynes/cm, respectively.

Reference herbal shampoo reduced the surface tension of water up to 31.504 dynes/cm as given in **Table 4** and shown in **Figure 5(a)**.

An increase in cleaning action was seen as the concentration of shikakai was increased in the shampoo formulations from F1 to F5. As given in **Table 4**, F1 formulation shows the lowest cleaning action (detergency power) with the value found as 24.30 % due to incorporated low amount of shikakai as 10 mL.

Whereas highest cleaning ability was recorded in case of F5 with the value found as 28.26 % when 18 mL of shikakai was used during preparation of formulation.

The reason is that shikakai act as good cleaning agent to remove dust and dirt particles entrapped in hair. High amount of cleaning agents resulted into higher cleaning ability.

The cleaning action was found to be 22.02 % when the test was conducted for reference herbal shampoo as shown in **Figure 5(b)**. The cleaning action of shampoo can also be interpreted in term of emulsion formation.

High percentage of emulsion formation indicates the better cleaning action of a shampoo formulation. Emulsion test was conducted for all the formulations and results were recorded as given in **Table 5**.

Emulsion volume was found in range of 45.5 - 59.5 % for different reported formulations as shown in **Figure 6**. This results also indicate that formation of emulsion volume was increased with increased use of shikakai contents.

Thus, cleaning action was increased with increase of emulsion volume. It was also observed that all the formulations showed solubility in water medium.

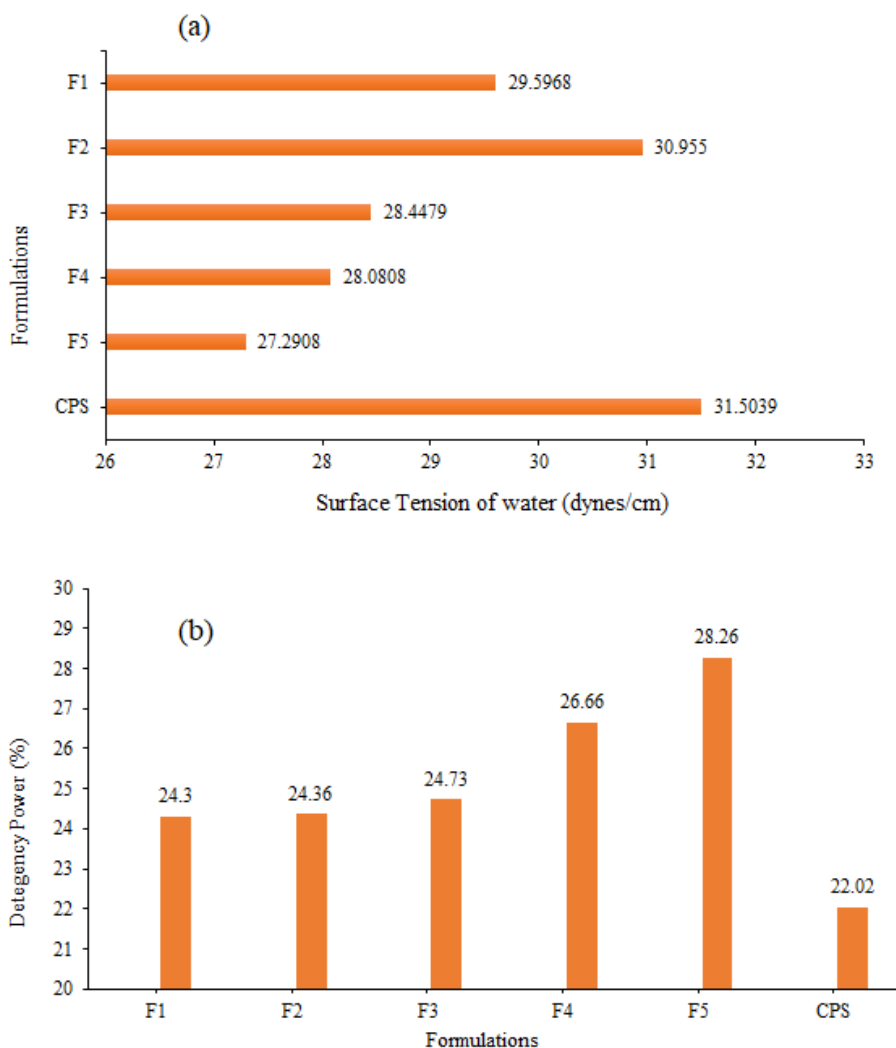


Figure 5: Effect of Different Shampoo Formulations on (a) Surface Tension of Water and (b) Cleaning Action

Table 5: Results of Solubility Test, Emulsion Test and Anti-Fungal Assessment

Formulations	Solubility	Emulsion (%)	Zone of inhibition
F1	Soluble in water	45.5	18 ± 0.06
F2	Soluble in water	49	19 ± 0.13
F3	Soluble in water	53.5	20 ± 0.05
F4	Soluble in water	56	23 ± 0.24
F5	Soluble in water	59.5	25 ± 0.29
CPS	Soluble in water	38	20.5±0.05

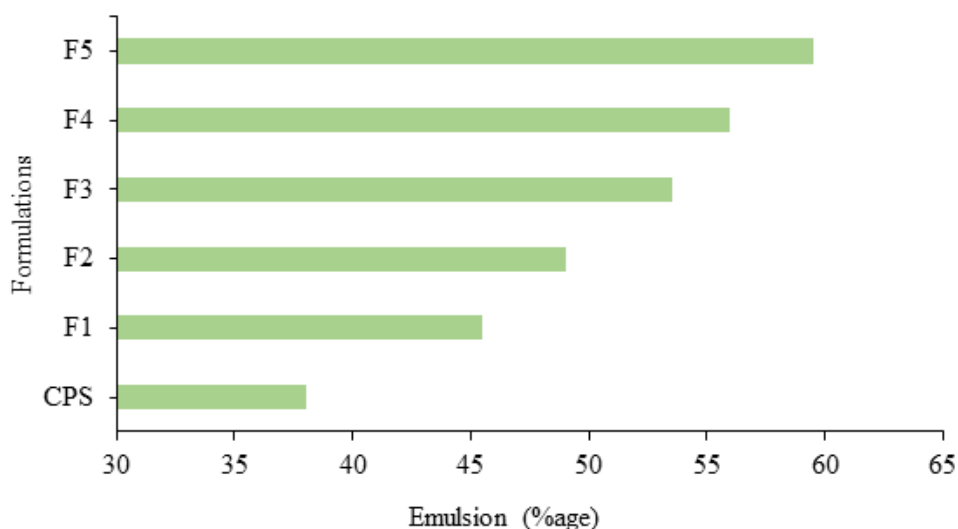


Figure 6: Emulsion Formation for Various Prepared Shampoo Formulations

Stability test performed for all formulations showed a slight increase in the pH of all formulations with the passage of time. Yet, it was observed that increased pH values were found in range of ideal values. Test also showed that the surface tension values were also increased but the change was very small. The foaming ability of the shampoo formulations was increased after two-week time period. This showed that the shampoo formulations were quite stable.

Anti-Fungal activity of various formulations was also tested. The diameters of the zone of inhibition were reported to be found in range of 25 to 18 mm as given in **Table 5**. This shows that the shampoo formulations produced had good anti-fungal properties. None of the prepared formulations showed any unwanted effects such as redness of skin, burning sensation or itchiness in the skin irritation test. All the formulations were found safe for use and did not show any damage or discomforting effects to the eyes in the eye irritation test. This reason is that shampoo is mainly composed of ingredients that are completely natural and do not cause any side effects.

4. CONCLUSION

Five different shampoo formulations were prepared by using neem, garlic or pomegranate peels as a potential antiseptic, anti-oxidant, antidandruff agent, collectively. Analysis of these formulations show that they did not have any adverse effects on the health of hairs, rather the anti-fungal activity increased dramatically. The shampoo formulations showed a zone of inhibition of 25 ± 0.29 mm which was even better than a polyherbal shampoo (control). Foam formation and stability test showed that F5 formulation indicates 175 and 180 mL foam formation in case of freshly prepared and two weeks old shampoo and even surpassed the values obtained in case of commercial shampoo used as standard. The

cleaning action of F5 depicted a detergency power of 28.26 % while commercial polyherbal shampoo showed the value 22.02 % that is low than F5 formulation. It was also seen that pH of the shampoo formulation remained stable overtime and in the ideal range, so it may not damage the hair.

Conflict of Interest

Authors declare no conflict of interest.

References

- 1) Devidas, P.V. and Hingne, L. Formulation and evaluation of herbal shampoo from piper betel and psidium guajava leaves, *International Journal for Research in Applied Science & Engineering Technology*, 10:6, 1-11 (2010)
- 2) Fazlolahzadeh, O. and Masoudi, A. Cosmetic evaluation of some Iranian commercial normal hair shampoos and comparison with new developed formulation. *International Journal of Pharmacognosy*. 2(5):259-65 (2015).
- 3) Rai, S. and Dasani, S. The Science of Shampoo: Herbal vs. Synthetic. *Srujan*. 49 (2014).
- 4) Saraf, S., Hargude, S.M., Kaur, C.D. and Saraf, S. Formulation and evaluation of herbal shampoo containing extract of *Allium sativum*. *Research Journal of Topical and Cosmetic Sciences*. 2(1):18 (2011).
- 5) Chandran, S., Vipin, K., Augusthy, A.R., Lindumol, K. and Shirwaikar, A. Development and evaluation of antidandruff shampoo based on natural sources. *Journal of Pharmacy and Phytotherapeutics*. 1(4):2321-5895 (2013).
- 6) Gubitosa, J., Rizzi, V., Fini, P. and Cosma, P.J.C. Hair care cosmetics: From traditional shampoo to solid clay and herbal shampoo, a review. 6(1):13 (2019).
- 7) Chandran, S., Vipin, K., Augusthy, A.R., Lindumol, K., Shirwaikar, A. Development and evaluation of antidandruff shampoo based on natural sources. *Journal of Pharmacy and Phytotherapeutics*, 1(4):2321-5895 (2013).
- 8) Revansiddappa, M., Sharadha, R. and Abbulu, K. Formulation and evaluation of herbal Anti-dandruff shampoo. *Journal of Pharmacognosy and Phytochemistry*. 7(4):764-7 (2018).
- 9) Pandey, A. and Tripathi, S. Concept of standardization, extraction and pre phytochemical screening strategies for herbal drug. *Journal of Pharmacognosy and Phytochemistry*. 2(5):115-9 (2014).
- 10) Chhouk, K., Uemori, C., Kanda, H. and Goto, M. Extraction of phenolic compounds and antioxidant activity from garlic husk using carbon dioxide expanded ethanol. *Chemical Engineering and Processing: Process Intensification*. 117:113-9 (2017).
- 11) Sithisarn, P., Supabphol, R. and Gritsanapan, W. Comparison of free radical scavenging activity of Siamese neem tree (*Azadirachta indica* A. Juss var. *siamensis* Valetton) leaf extracts prepared by different methods of extraction. *Medical Principles and Practice*. 15(3):219-22 (2006).
- 12) Al Badi, K. and Khan, S.A. Formulation, evaluation and comparison of the herbal shampoo with the commercial shampoos. *Beni-Suef University Journal of Basic and Applied Sciences*. 3(4):301-5 (2014).
- 13) Saad, A.H., Kadhim, R.B. and Rasool, B.A. Formulation and evaluation of herbal shampoo from *Ziziphus spina-christi* leaves extract. *International Journal of Research in Ayurveda & Pharmacy*. 2(6):1802-6 (2011).

- 14) Mainkar and Jolly. Evaluation of commercial herbal shampoos. *International journal of cosmetic science*. 22(5):385-91 (2000).
- 15) Lodha, G. Formulation and evaluation of polyherbal shampoo to promote hair growth and provide antidandruff action. *Journal of Drug Delivery and Therapeutics*. 9(4-A):296-300 (2019).
- 16) Naeem, A., Saddique, S. and Chand, S.A. Advancement and Future Directions towards Herbal Treatment for Various Diseases. *Saudi Journal of Medical and Pharmaceutical Sciences*, 331-341 (2019).
- 17) Pradhan, A. and Bhattacharyya, A.J.J.S.S.T. Shampoos then and now: synthetic versus natural. *Journal of Surface Science and Technology*30:59-76 (2014).
- 18) Malpani, T., Jeithliya, M., Pal, N. and Puri, P. Formulation and evaluation of Pomegranate based herbal shampoo. *Journal of Pharmacognosy and Phytochemistry*. 9(4):1439-44 (2020).
- 19) Al Badi, K., Khan, S.A. Formulation, evaluation and comparison of the herbal shampoo with the commercial shampoos. *Beni-Suef University Journal of Basic and Applied Sciences*, 3(4):301-5 (2014).
- 20) Gahlawat, J., Sharma, D., Thakur, G.S., Chobdar, J. and Sharma, V. Formulation and evaluation of polyherbal liquid shampoo. *European Journal of Biomedical and Pharmaceutical Sciences EJBPS*. 6(7):149-54 (2019).
- 21) Mainkar, Evaluation of commercial herbal shampoos. *International journal of cosmetic science* 22(5):385-91 (2000).