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CHARACTERIZATION AND PERFORMANCE METRICS OF A NOVEL POLYHERBAL SHAMPOO CONTAINING BIOACTIVE COMPONENTS

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Abstract

Shampoo is a cosmetic formulation primarily used for cleansing the hair and scalp. In the current market, most commercially available shampoos are synthetically derived. The objective of this study is to develop a herbal shampoo using natural plant-based ingredients that exhibit comparable benefits to those offered by chemical formulations, such as promoting hair growth, enhancing shine, improving hair strength, and providing anti-dandruff effects. The key herbal components incorporated into this polyherbal formulation include extracts from *Terminalia chebula*, *Acacia concinna* (Wild.), *Lawsonia inermis*, *Trigonella foenum-graecum*, *Phyllanthus emblica*, *Sapindus mukorossi*, *Cyamopsis tetragonolobus*, *Citrus sinensis*, *Moringa oleifera*, and *Camellia sinensis*. These botanicals contribute various functional properties to the shampoo, such as natural gelling, emulsifying, moisturizing, hydrating, surfactant, thickening, and foaming effects. Multiple formulations were prepared and assessed in a laboratory. They were then evaluated against several physicochemical parameters, including organoleptic characteristics, pH, dirt dispersion, surface tension, foaming capacity, and formulation stability, and compared with standard synthetic shampoos available in the market. The study successfully resulted in the development of a high-quality, cost-effective herbal shampoo that may serve as a superior alternative to conventional chemical-based products.

Keywords: Herbal Shampoo, Organoleptic, Phytoconstituents, Physicochemical Characterization.

1. INTRODUCTION

Hair is protein in nature and essential part of human body that originates from the skin follicles. It is the essential part of beauty derived from the ectoderm and acts as a protective covering on the body. It consists of three parts: root, stem and bulb with cortex, cuticle as layers and medulla. Cuticle provides smoothness and shine whereas cortex gives strength [1]. People are using herbs since early times for hair cleansing and beautifying whereas for today shampoo is the most generally utilized product for this purpose. In 1500, it was originated by blending herbs in India. The "Shampoo" word comes from the Hindi word "Champo" which means "to soothe". It evolved into commercial products between 1903-1927 [2]. These are actually a solution of detergents

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along with cleansers and other ingredients; which may be synthetic or herbal. Synthetic shampoos have high foaming and cleansing ability but they can also show some side effects after regular use like eyes irritation, dryness, and hair loss due to chemicals like Coamidopropyle betaine and sodium lauryl sulfate. Other ingredients include polyethylene glycol (PEG), polysorbates, phenoxyethanol, and potassium sorbate which may cause long term health issues and disrupt pH balance. Herbal formulations can replace synthetic once as they have negligible side effects, environment friendly, promote hair health and have low cost [3].Current polyherbal shampoo contains henna (*Lawsonia inermis*), amla (*Phyllanthus emblica*), fenugreek (*Trigonella foenum-graecum*), shikakai (*Acacia concinna*), guar gum (*Cyamopsis tetragonoloba*), reetha (*Sapindus mukorossi*), spinach (*Spinacia oleracea*) and suhanjana (*Moringa oleifera*) due to their dandruff fighting, hair conditioning and growth promoting properties. *Acacia concinna,* included in it is a superb cleaner for hair and does not remove natural oils from hair and scalp and also helps to balance scalp pH. Similarly, *Phyllanthus Emblica* acts as a hair conditioner and provides nutrients. *Trigonella foenum graecum* is a source of protein and helps in hair growth. *Hibiscus* is used as growth promoter and to treat hair fall and dandruff [4]. The plants extracts used in the formation of polyherbal shampoo are extracted through decoction, infusion, maceration, and soxhlet extraction methods. For slightly water-soluble extracts, soxhlet method is suitable which is a continuous hot process, while decoction is suitable for water-soluble materials as it involves hot extraction with water. These methods ensure efficient extraction without heat damage to the antioxidants and both methods are used widely in shampoo formulations. Shampoos for both oily and dry hair usually use same concentration of detergent with variety in other ingredients. A source of comparison is a commercially available herbal shampoo having similar ingredients as used in the present formulation [5].

2. MATERIALS AND METHODS

2.1 Chemicals

Methyl paraben, citric acid, guar gum, and Lavender oil

2.2 Collection and Drying of Herbs

Some of the plants were obtained in dried form from the local market of Thokar Niaz Baig, Lahore on Oct 15, 2021. These plants include *Terminalia chebula* (harada), *Camellia sinensis* (green tea) and *Murraya koenigii* (curry patta). They were authenticated by a certified botanist from University of Central Punjab, Lahore, Pakistan. Other ingredients like *Moringa oleifera* (sohanjna), *Sapindus mukorossi* (reetha), *Phyllanthus emblica* (amla), *Acacia concinna* (shikakai), *Lawsonia inermis* (henna), *Citrus sinensis* (orange peel) and, *Trigonella foenum* (fenugreek) were obtained in dried form from the local market, Thokar Niaz Baig, Lahore on Oct 10, 2024. *Rosa species (*Rose) were sourced from a local plant nursery on Oct 25, 2024. A polyherbal shampoo was also sourced from superstore for comparative analysis. The herbs were dried by first cleaning them under

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running tap water, then placing them under shade after wrapping with a cotton cloth to prevent dust. Dried leaves were subjected to various extraction processes.

2.3 Preparation of Herbal Extract

To prepare the herbal extracts of the selected plants, Decoction, Maceration & Soxhelt extraction methods were used. In decoction, the dried plants were taken and converted into coarse powder by using grinder (ND National Grinder JB-222). This powder was sieved using a 100 Mesh sieve. In a pre-washed, pre-dried 500 ml beaker, 1:4 sieved powder and distilled water were mixed. It was constantly boiled until only one fourth of the solution remained. Allowed it to cool for a few minutes before filtering then filtered it using Whatsman filter paper number 1. After cooling, beaker was covered with aluminum foil and stored it in the refrigerator until further analysis [6]. For maceration process, 70 % ethanol was used as solvent. Dried leaves of green tea were ground into powder and this powder was added into a glass beaker having ethanol solution in it. A ratio of about 1:10 leaves to ethanol was used for extraction. The liquid extract was filtered using Whatsman filter paper number1 and then concentrated by rotary evaporator at temperature of 60°C. It was cooled and covered with aluminum foil and placed in refrigerator until further treatment [7]. In Soxhelt method, the powdered sample was placed in a porous thimble in the extraction chamber. Water was used as solvent. The solvent to sample ratio was 15:1. The solvent temperature was kept at 100° C for nearly two hours. The extraction was continued until the round bottom flask contained all of the soluble components. After that, the extract was concentrated by evaporation and then preserved afterwards [8].

2.4 Formulation of Herbal Shampoo

The mass of dried plants, powdered herb and the weight of extracts is given in table 1.

Table 1: Composition of Herbal Shampoo Formulations

Ingredients	F1(mL)	F2(mL)	F3(mL)	F4(mL)	F5(mL)
Harada	1.5	2	2.5	3	3.5
Shikakai	4	3.5	3	2.5	2
Amla	2	2.5	3	3.5	4
Reetha	1.5	2	2.5	3	3.5
Henna	2.5	2	1.5	1	0.5
Tea	2.5	2	1.5	1	0.5
Orange peel	1	1	1	1	1
Moringa	1.5	2	2.5	3	3.5
Fenugreek	3.5	3	2.5	2	1.5
Curry patta	3	2.5	2	1.5	1
Rose	2	2.5	3	3.5	4
Gur Gum	q.s				
Methyl Paraben	q.s				
Citric acid	q.s				
Lavender oil	q.s				

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2.5 Physiochemical Analysis of the Formulations

Several analyses were conducted to evaluate the prepared herbal shampoo formulations. The formulations were evaluated for odor, texture, color, and taste as shown in Figure 1(a). All samples were observed for physical appearance followed by other analysis.

In the dirt dispersion test, two drops of herbal shampoo were mixed with 10 ml of distilled water in a falcon tube and shaken well. One drop of blue ink was added to the mixture and kept undisturbed for 10 minutes. The ink was seen settling at the bottom of the tube, while some quantity remained suspended in the liquid. The amount of ink was assessed as none, light, moderate, and heavy as illustrated in Figure 1(b) [9].

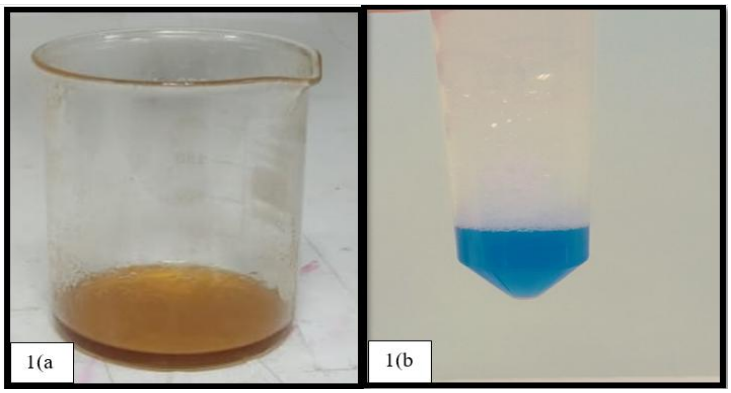
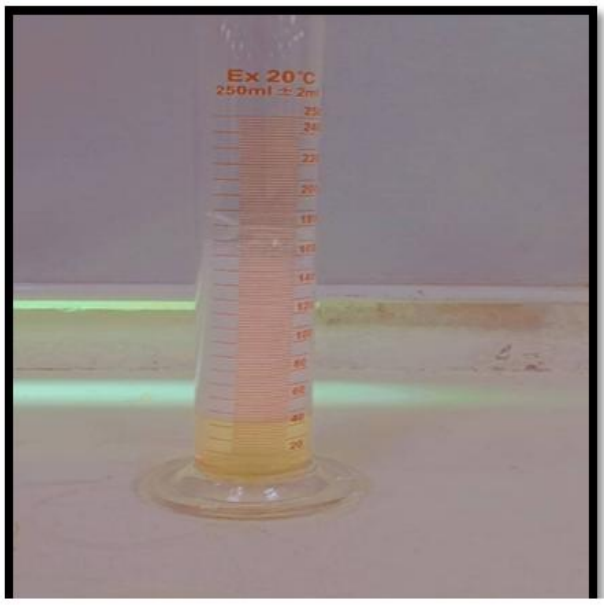


Figure 1: (a) Organoleptic Property, 1(b) Dirt Dispersion Test



1(c) Foaming Ability and Stability Test

To assess the stability of herbal formulations, samples were stored for 2 weeks at 25–30 °C. Before storage, initial parameters such as color, pH, solid contents, cleaning action,

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surface tension, foaming ability, and dirt dispersion were measured. After the storage period, the same tests were repeated to evaluate physical and chemical stability [10].

A skin irritation test was also performed. A small amount of the herbal formulation was applied on the back of a volunteer's elbow and left for 5 minutes. The area was observed for any signs of redness or irritation [11].

The foaming ability and foaming stability were assessed using the cylinder shake method. For this purpose, 1% shampoo solution (50 ml) was poured into a 250 ml graduated cylinder and shaken for 10 minutes as shown in Figure 1(c). Foam volume was recorded after 1 minute and then at 1-minute interval for up to 4 minutes [12].

To determine the percentage of solid contents, 8 g of herbal shampoo was placed in a pre-weighed evaporating dish and weighed. The shampoo was evaporated using a hot plate until complete drying. The dish was weighed again, and the percentage of solids was calculated using the following formula:

$$\% \text{ of solid contents} = \frac{\text{Weight of dried shampoo}}{\text{Initial weight of the shampoo}} \times 100$$

Surface tension of the shampoo solution was measured by the stalagmometer drop count method. A 10 ml shampoo solution was diluted in 90 ml distilled water. The flat end of the stalagmometer was dipped in the solution and filled up to a marked point. Drops falling from point A to point B were counted as shown in Figure 2.

Figure 2: Surface Tension determination by Stalagmometer

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The following formula was used:

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

Where W_1 is the weight of the empty beaker, W_2 the weight with distilled water, W_3 the weight with shampoo solution; N_1 and N_2 are the number of drops of water and shampoo respectively, and R_1 is the surface tension of water [13]. Viscosity was measured after cleaning and drying the viscometer. Shampoo solution was made by mixing 10 ml shampoo in 90 ml distilled water. Bulb 1 of the viscometer was filled, and the liquid was allowed to flow from mark A to B. The time taken was recorded. This was repeated for both water (t_1) and shampoo (t_2). The viscosity was calculated as:

$$\boldsymbol{\eta_1} = \frac{\boldsymbol{\rho_1 t_1}}{\boldsymbol{\rho_2 t_2}} \times \boldsymbol{\eta_2}$$

Where η_1 and η_2 are viscosities of water and sample, and ρ_1 and ρ_2 are densities of water and shampoo [14]. Wetting time test was carried out by placing a one-inch canvas paper disc on a 1% shampoo solution, smooth side down. The time taken by the disc to sink completely was measured with a stopwatch as shown in Figure 3 [15].

Figure 3: Wetting Time Test

pH of each sample was measured using a pH meter (HI9813-6). A 5% v/v solution was prepared by dissolving 5 ml shampoo in 45 ml distilled water. The pH meter was calibrated before inserting the probe into the sample solution [16].

For the cleaning action test, 1 g shampoo solution in distilled water was prepared in a flask. A grease-covered yarn was dipped into it and the flask was shaken 50 times per minute for 4 minutes as shown in Figure 4.

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Figure 4: Cleaning Action Test

After drying, the yarn was weighed. Detergency power was calculated using the formula:

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

Where DP is detergency power, T is weight of washed sebum, and C is weight of unwashed sebum [17].

In the emulsion test, 1 ml of shampoo was mixed with 1 ml of coconut oil in a test tube, while a blank test tube had 1 ml water with 1 ml oil. The tubes were vortexed for 30–60 seconds and left for 24 hours. The emulsion height was measured and calculated using:

$$\textbf{Percentage Emulsion} = \frac{\textbf{Height of emulsion}}{\textbf{Total height of sample}}$$

Solubility was tested by mixing 2 ml shampoo with 100 ml distilled water. The mixture was shaken and heated, then cooled after 10 minutes. Any residue was noted as shown in Figure 5 [18].

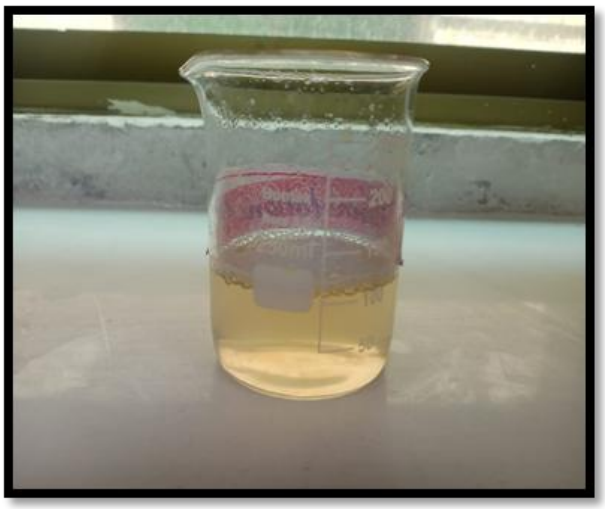

Figure 5: Solubility Test

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3. RESULTS AND DISCUSSION

Plants samples were collected and verified according to standard procedures. In figure 6 the dry and fresh weights of various plant materials along with the percentage of water content used in the formulation are given. For example, moringa leaves had a dry weight of 380 g and a fresh weight of 1200.01 g, producing water content of 30.66%. Henna leaves, dried down to 240.10 grams producing a water content of 23.01% a fresh weight of 999.99 grams. This shows the extractable material which is left after drying. Studies shows moringa and henna retains nutrients in the hair, antifungal and is beneficial for people with dandruff, hair loss, and other microbial problems.

3.1 Characterization of Shampoo Formulations

Different herbal shampoo formulations were prepared using plants that contains important herbal ingredients beneficial for hair. All quality control tests were performed and compared with commercially available shampoo. The results obtained from evaluation of different parameters showed that plants used in herbal formulations gave more suitable and acceptable results than commercial shampoo. Both the herbal formulations and marketed shampoo were studied with respect to their color, odor and clarity. Results for organoleptic properties, dirt dispersion, skin irritation and solubility of different formulations are presented in table 2. The results show all of the formulations (F1-F5) have a moderately pleasant odor, dark to light brown in color, bitter in taste , smooth in texture, soluble in water, moderate dirt dispersion and have no harmful effect on skin . While the commercial shampoo which was taken for reference had a noticeable pleasant smell, green in color, soluble in water, moderate dirt dispersion and have no harmful effect on skin as well.

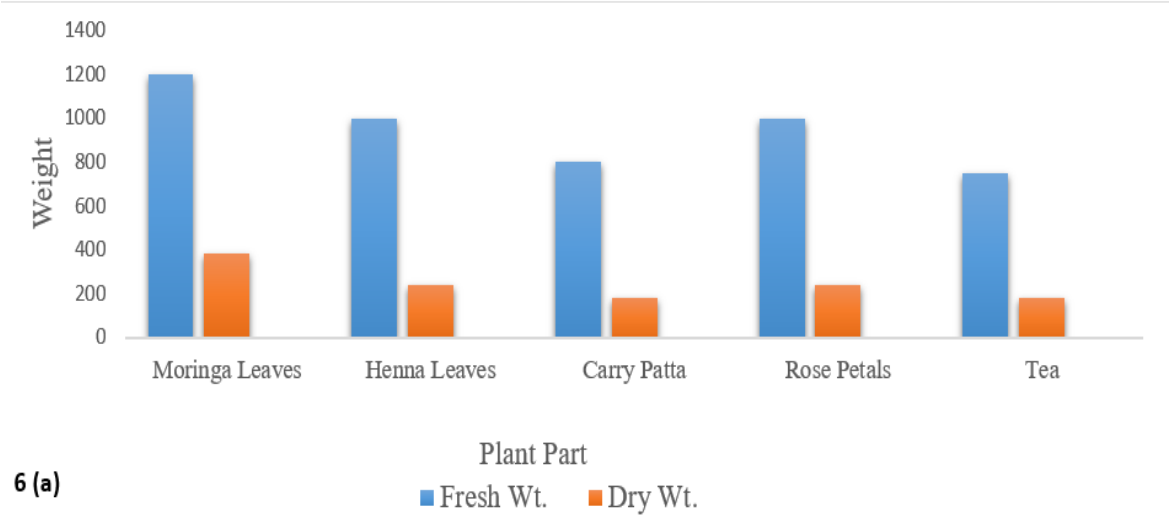

Figure 6: (a) Graphical representation of fresh weight and dried weight,

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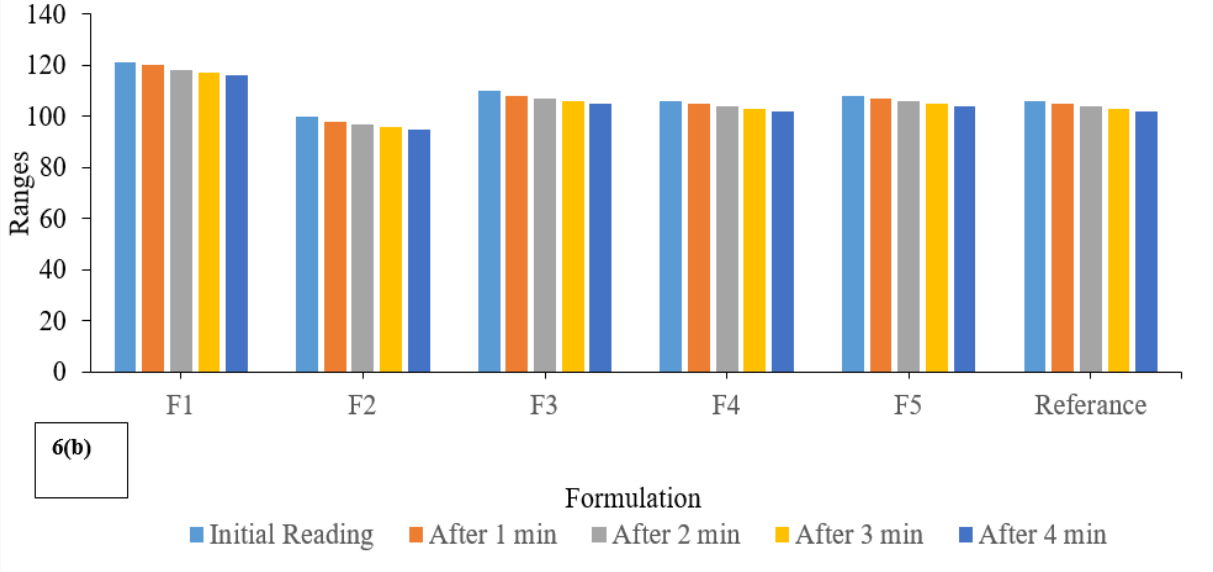

Figure 6: (b) Graphical representation of Foaming ability

Table 2: Organoleptic Analysis, Dirt Dispersion, Skin Irritation and Solubility

Sr. No	Organoleptic Evaluation	F1	F2	F3	F4	F5	Reference
1	**Color**	Light brown	Light brown	Dark brown	Dark brown	Dark brown	Green
2	**Odor**	Slightly pleasant	Slightly pleasant	Slightly pleasant	Slightly pleasant	Slightly pleasant	Pleasant
3	**Taste**	Bitter	Bitter	Bitter	Bitter	Bitter	Bitter
4	**Texture**	Smooth	Smooth	Smooth	Smooth	Smooth	Smooth
Dirt dispersion		Moderate	Moderate	Moderate	Moderate	Moderate	Moderate
Skin irritation		No harmful effect on skin	No harmful effect on Skin	No harmful effect on Skin	No harmful effect on Skin	No harmful effect on Skin	No harmful effect on Skin
Solubility		Soluble in water	Soluble in water	Soluble in water	Soluble in water	Soluble in water	Soluble in water

pH plays a key role in improving the hair quality and reducing eye irritation. It also helps to reduce damage to hair. Higher pH can damage the hair cuticles and mild acidic pH gives shine to hair and promotes tightening of scales. It is reported that the ideal pH ranges between 5-7 and the pH of commercial shampoo (Reference) was observed 6.7. Out of five formulations the highest pH was shown by F1 and F2 i.e 5.6 which is shown in Table 3. The graphical representation of pH in figure 7(a). The viscosity of shampoo plays a key role in determining stability of shelf life, ease of its flow and its application on hair. The viscosity of herbal shampoo was 46.6 millipoise (average) which is good enough for hair [18]. Whereas the reference formulation showed 48 millipoise. F5 out of five

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prepared formulations depicted excellent viscosity i.e 50 milliposie as shown in Table 3 and Figure 7 (b).

Table 3: Evaluation of Formulations for pH, Solid Content (%), Wetting Time (sec), Cleaning Action, Surface Tension and Viscosity

Formulation	pH	Solid Content (%)	Wetting Time (sec)	Cleaning Action	Surface Tension	Viscosity (Millipose)
F1	5.66 ± 0.05	22.51 ± 0.03	124.23 ± 0.3	22.08 ± 0.12	32.07 ± 0.11	45
F2	5.66 ± 0.05	21.00 ± 0.09	121.10 ± 0.1	21.24 ± 0.08	35.06 ± 0.12	47
F3	5.54 ± 0.06	23.72 ± 0.02	124.45 ± 0.3	25.18 ± 0.24	34.39 ± 0.53	43
F4	5.33 ± 0.01	27.026 ± 0.05	124.2 ± 0.3	30.48 ± 0.33	31.36 ± 0.72	47
F5	5.33 ± 0.01	24.51 ± 0.04	122.8 ± 0.7	26.15 ± 0.15	32.18 ± 0.27	50.28
Reference	6.7 ± 0.11	9.50 ± 0.01	84 ± 0.2	28.56 ± 0.34	35.95 ± 0.45	48.78

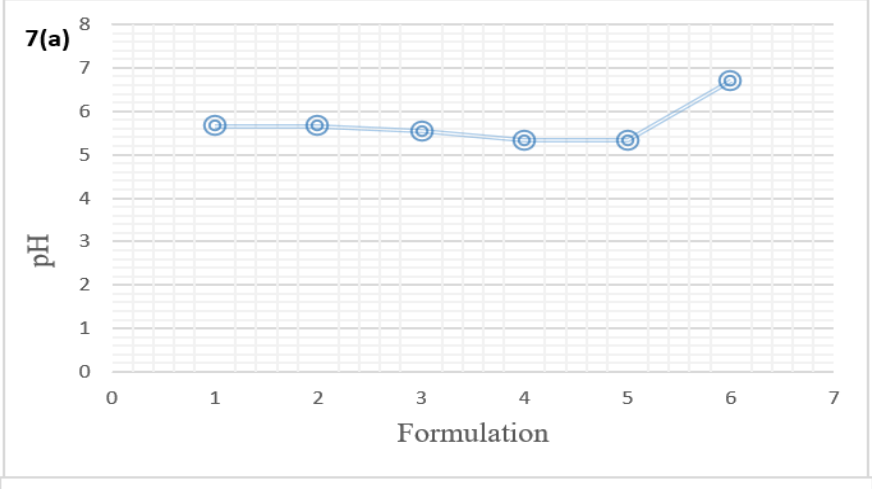

Figure 7: (a) Graphical representation of pH,

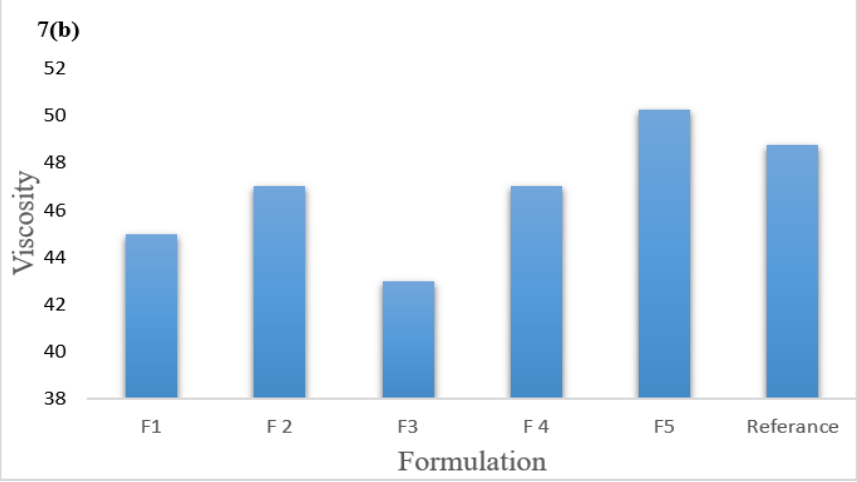

Figure 7: (b) Graphical representation of Viscosity.

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Meduri *et al.* have reported that ideal shampoos should have solid content in the range of 20-30% [19]. If the solid content is very low, it is difficult to apply on hair and if it is very high then hard to wash it out.

The percentage solid content of commercial herbal shampoo (Reference) was 9.5% while the formulated shampoos were found to have solid contents in the range of 15-30% i.e within the ideal range but the best results of percentage of solid content was shown by F4 [shown in Table 3 and its graphical representation in Figure 8(a)].

Wetting time of a formulation depends on the concentration of surfactant, surface tension and diffusion factor. The main purpose of wetting agent is to reduce surface tension. It helps to improve its efficacy.

Wetting ability of formulations was evaluated by using canvas disc method. Wetting time range should be 4-240 seconds. Lesser the time means more wetting efficacy. The commercial shampoo (Reference) has wetting time about 84 seconds while the prepared formulations presented wetting time in the range of 122-124 seconds which is also an acceptable characteristic.

The best result was shown by F2 which is represented in Table 3 and its graphical representation in Figure 8(b).

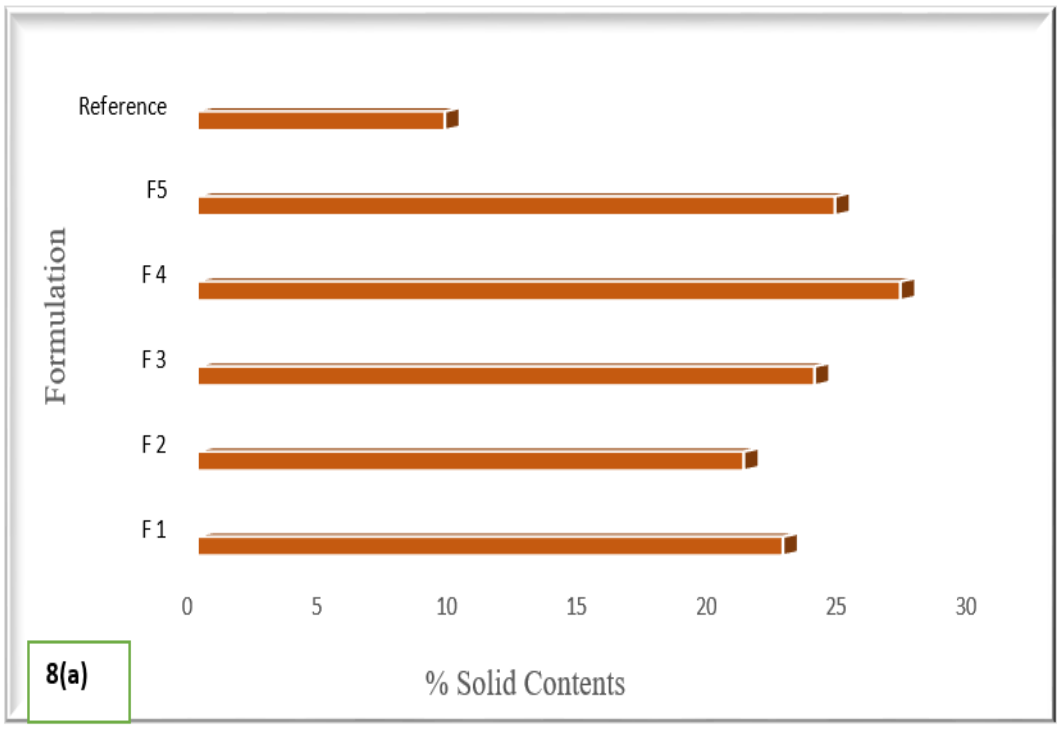

Figure 8: (a) Graphical representation of Solid Contents

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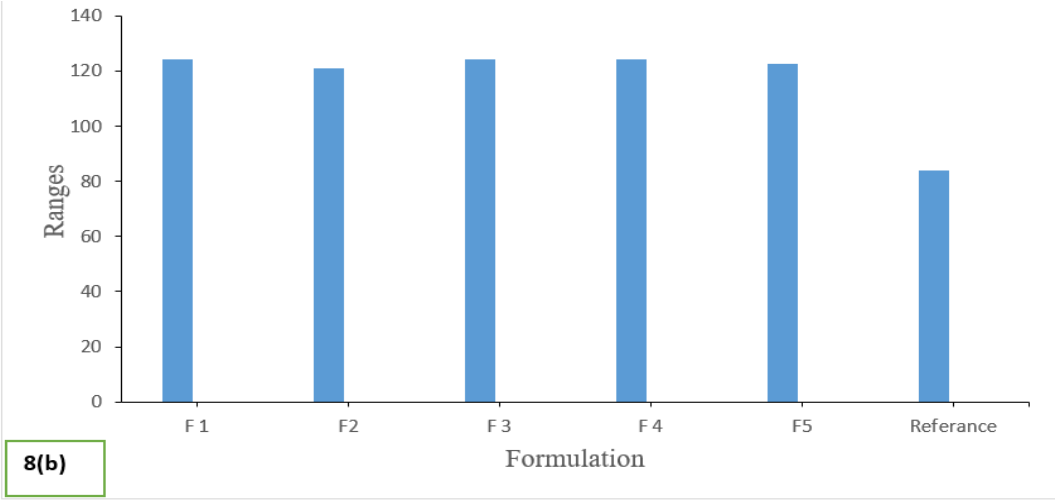

Figure 8: (b) Graphical representation of Wetting Time Test

Foaming ability is also an important factor to evaluate shampoos ideality for hair. Foam is formed due to the presence of surfactants in the shampoo. When there is high volume of foam, it is considered as high-quality product and becomes the preference of consumer.

Commercial shampoo (Reference) has foaming ability in the range of 102-106 ml. The formulated herbal shampoos have foaming ability in the range of 102 -121ml and remained stable for few minutes *i.e.* the ideal range showing good quality of shampoos. The highest foaming ability was shown by F1 which is 121ml as shown in Table 4.

Dirt dispersion test helps to evaluate the cleansing action of formulations, if ink remains in the foam, it shows poor quality of shampoo. When dirt remains in foam, it is difficult to rinse off and will deposit on the hair [14]. The formulated shampoos have very light ink left in the foam which reflects its excellent cleansing action.

Table 4: Foaming Ability of shampoo formulations

Time (min)	Foam Volume					
	F1(ml)	F2(ml)	F3(ml)	F4(ml)	F5(ml)	Reference(ml)
Initial Reading	121	100	110	106	108	106
After 1 min	120	98	108	105	107	105
After 2 min	118	97	107	104	106	104
After 3 min	117	96	106	103	105	103
After 4 min	116	95	105	102	104	102

The shampoo should have the ability to decrease the surface tension of water up to 40 dynes/cm which leads in improving detergency of shampoo. When the surface tension is less it means shampoo has good cleansing ability. It is reported that the ideal surface tension range is 31-38 dyne/cm [2]. The Reference formulation has surface tension 35 dyne/cm while the best surface tension was shown by F2 that is about 35 dynes/ cm which is shown in Table 3 and in Figure 9(a).

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Cleansing action test was performed on wool yarn. Cleansing action is the removal of sebum which is an important function of shampoo. It is reported that the cleansing of herbal shampoo was 21% [14].

The reference formulation has 28% cleansing action while out of five formulations excellent results was shown by F4 that is about 30% [shown in Table 3 and its graphical representation is presented in Figure 9(b)]

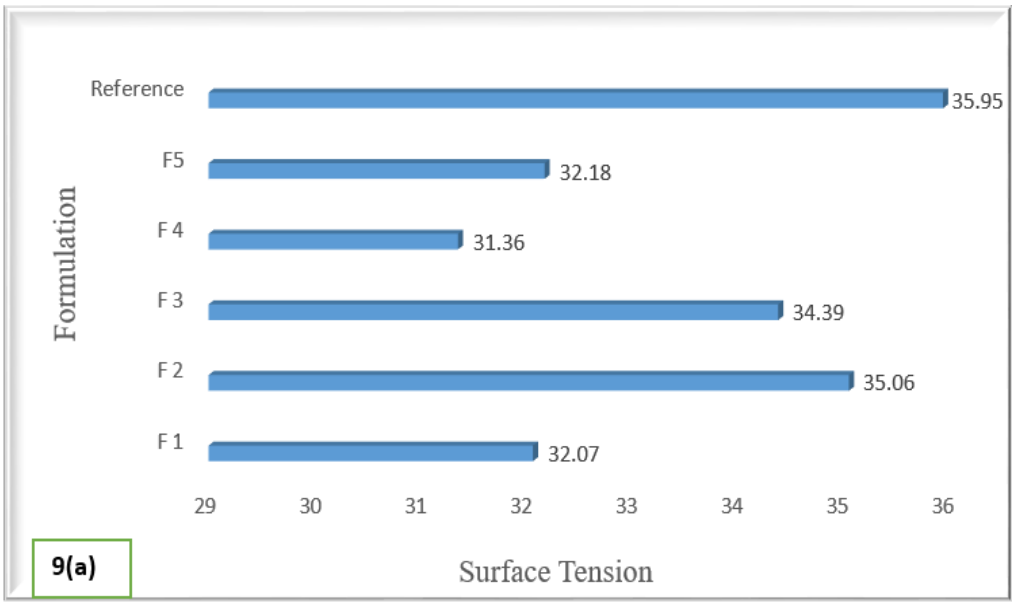

Figure 9: (a) Graphical representation of Surface Tension,

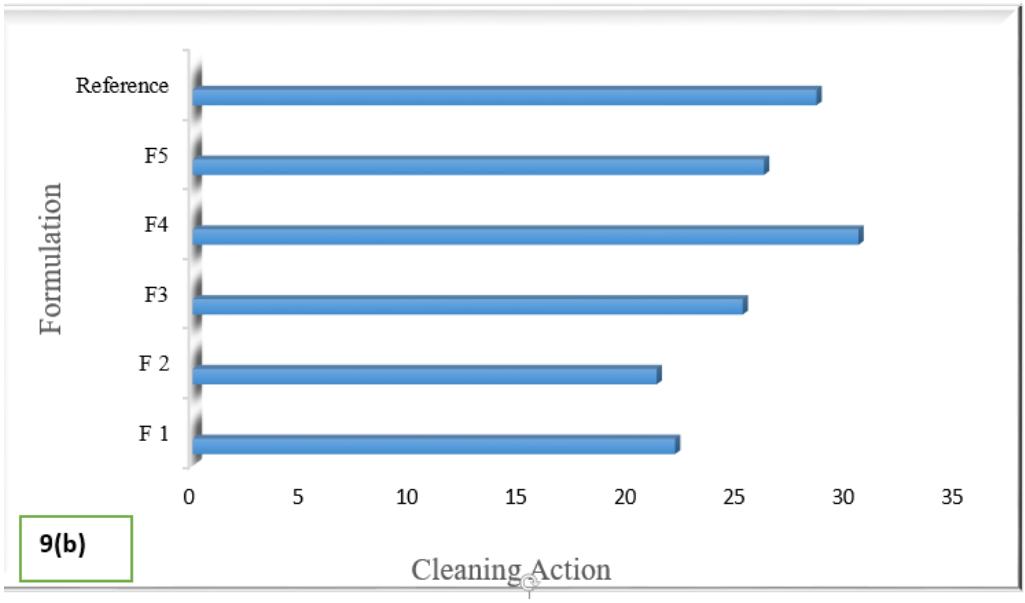

Figure 9: (b) Graphical representation of cleaning Action

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Skin irritation test showed that formulation have no effects on skin and this is due to the absence of harmful ingredients. Inflammation or irritation mostly caused by synthetic chemicals. But in these formulations all herbal ingredients were present. Prepared herbal formulations and reference (commercial shampoo) both showed no irritation which means these are harmless and good quality formulations.

Stability test indicates that formulations are physically and chemically stable during the storage period. Both the reference and prepared herbal formulations remained stable during storage period and there was no change in the organoleptic properties of formulations.

High percentage of emulsion formation indicates the better cleaning action of a shampoo formulation. Emulsion volume was found in range of 49 - 60 % for different reported formulations as shown in Figure 10. This results also indicate that F3 showed the maximum percentage of emulsion showing its highest cleaning ability.

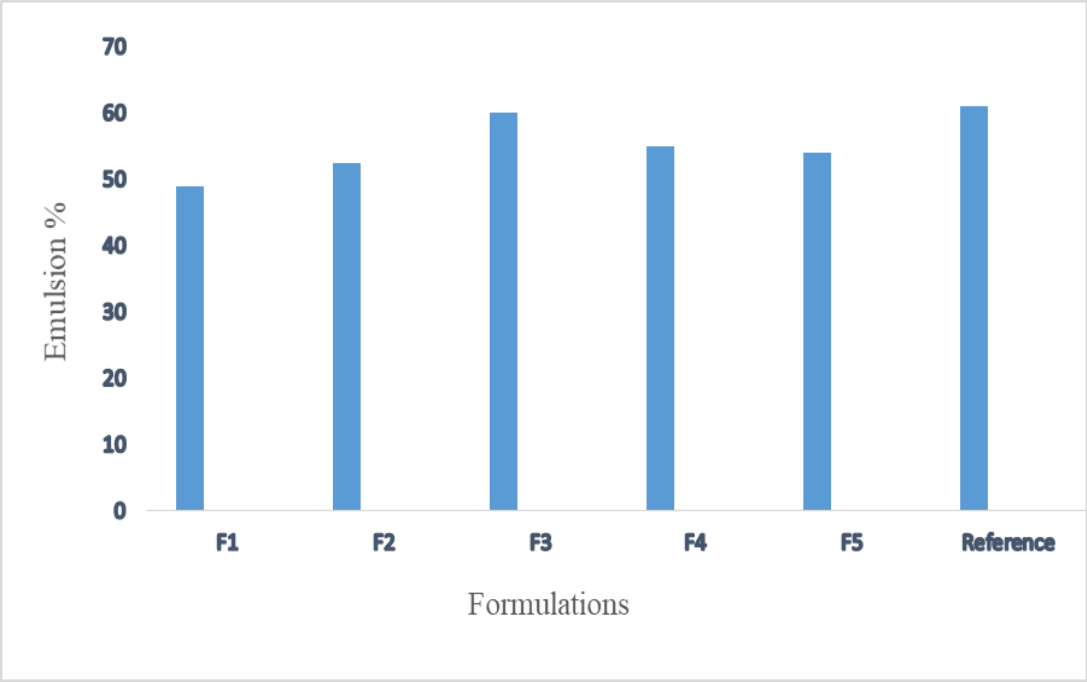

Figure 10: Graphical representation of Emulsion Test

4. CONCLUSION

The purpose of the study is to develop a stable and effective herbal shampoo that can replace synthetic shampoos. Different herbal formulations are prepared, compared and evaluated with commercially available shampoo in terms of various parameters including pH, surface tension, foaming ability, viscosity, percentage solid content, cleansing action, wetting time and stability *etc*.

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It is concluded form the results that the formulated herbal shampoos are as good as commercially available synthetic shampoo and, in some parameters, even much safer than synthetic one. Herbal formulations are additionally eco-friendly. It also reveals that local medicinal plants have a lot of promise for producing high-quality shampoo.

A more radical approach in popularizing herbal shampoo would be to change the consumer expectations from a shampoo, with emphasis on safety and efficacy. The onus is on the formulators to influence customer regarding what constitutes a good shampoo.

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