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FORMULATION AND EVALUATION OF HERBAL SHAMPOO

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Abstract:

The present study aims to formulate and evaluate a herbal shampoo incorporating multiple natural ingredients known for their therapeutic and cosmetic benefits. The formulation includes Shikakai, Aloe vera, Neem, Hibiscus, and Black sesame, combined with suitable excipients to enhance stability and usability. These herbs were selected for their traditional efficacy in promoting hair growth, improving scalp health, providing natural cleansing, and exhibiting antimicrobial and conditioning properties. The shampoo was prepared through standardized procedures and evaluated for key physicochemical parameters including pH, viscosity, foam stability, dirt dispersion, wetting ability, solid content, and surface tension. All prepared formulations exhibited desirable characteristics suitable for cosmetic use. Among them, one formulation demonstrated superior performance in terms of cleansing efficiency, consistency, aesthetic appeal, and stability. The findings support the use of herbal constituents in developing safe, effective, and eco-friendly alternatives to conventional synthetic shampoos.

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INTRODUCTION:

Hair is regarded as an essential component of human attractiveness, humans have used herbs for cleansing, beautifying, and hair management since ancient times [1].

Shampoos are used not only to clean hair but also to keep it lustrous, thick, longer, and softer while also eliminating oil. There are several varieties of shampoos available, including medicinal shampoo, liquid herbal shampoo, solid gel shampoo, powder shampoo, and clear liquid shampoo lotion [2,3]. Keratin is the primary ingredient in hair. One amazing protein that resists deterioration is keratin. Synthetic agents have dominated the formulation industries for many years, but people are now drawn to natural products because they are less costly and have fewer negative effects than synthetic ones, which can harm skin, hair, and eyes [4].

The essential component of human attractiveness is hair. Since ancient times, people have used herbs to clean, adorn, and manage their hair. Synthetic substances have risen in popularity throughout time, but people are now more conscious of their negative effects on skin, eyes, and hair. Because they are less costly and have less negative effects, the community in these areas is drawn to herbal goods. Shampoos and hair cleansers are used for more than just cleaning; they also give hair a glossy finish and keep it manageable and greasy.

There are many different kinds of shampoos, including lotion shampoo, solid gel shampoo, powder shampoo, clear liquid shampoo, medicinal shampoo, liquid herbal shampoo, and more. Regarding the stability standards for herbal shampoos. Simple or basic shampoo, antibacterial or antidandruff shampoo, and nutritious shampoo with vitamins and amino acids are all possible depending on the components a protein hydrolyses [5]. In terms of their morphological, anatomical, and biochemical characteristics, the qualitative and quantitative parameters of various herbal medications are distinct and can be a useful tool for differentiating drug samples. Significant information on the composition, alterations, developmental phases, and tissue differentiation of the cell wall may be obtained by combining histochemistry with microscopy.

One of the most objective techniques that may be effectively used in a variety of fields, including gene expression research, phytoconstituent localisation studies, secretory structure characterisation, pathogen invasion mechanism investigations.

METHODS & MATERIALS:

Table 1: **Methods and materials Used for formulation and evaluation of herbal shampoo**

Category	**Details**
Formulation Type	**Herbal Shampoo (Batches 1 to 7)**
Active Ingredients	Shikakai, Aloe vera, Neem, Hibiscus, Black sesame
Preservative	Methyl Paraben (0.5 ml per batch)
Additives	Rose Water (1 ml per batch), Distilled Water (Q.S. to adjust volume)
Batch Variations	Each batch differs in the quantity of active ingredients (3–5% variations)
Equipment Used	Beakers, Stirrer, pH meter, Viscometer, Graduated Cylinders, Stopwatch
Method of Preparation	Mixing of herbal powders/extracts with aloe vera gel and additives in sequence; stirred until uniform consistency achieved
Storage Condition	Stored in airtight containers at room temperature
Evaluation Parameters	pH, Viscosity, Foaming ability, Wetting time, Solid content, Surface tension, Dirt dispersion, Skin irritation test, Cleaning ability, Stability study
Graphical Tools Used	Bar graphs created using matplotlib to compare pH, viscosity, foaming, and stability

Methods of Formulation for Seven Batches of Herbal Shampoo

The development of herbal shampoo formulations involves careful selection, proportioning, and mixing of herbal and auxiliary ingredients to ensure optimal cleansing, foaming, conditioning, and scalp care properties. Each of the seven batches in this study was formulated using a systematic and reproducible method, with minor variations in herbal extract quantities to evaluate their impact on product quality. The base methodology remained consistent across all batches to maintain uniformity in preparation while enabling comparative evaluation.(5)

1. **Preparation of Herbal Extracts:**

The primary herbal ingredients used include Shikakai, Aloe vera, Neem, Hibiscus, and Black Sesame. Each component was either procured as dried powder or freshly harvested and processed into paste or juice using standard extraction methods.

Shikakai (Acacia concinna): Dried pods were crushed into powder and sieved through a mesh. The powder was then soaked in warm distilled water for 4–6 hours and filtered using muslin cloth to obtain a saponin-rich extract.

Aloe vera (Aloe barbadensis): Fresh Aloe vera leaves were washed, and the outer green rind was peeled to collect the inner gel. The gel was homogenized using a blender to obtain a smooth extract.

Neem (Azadirachta indica): Fresh neem leaves were boiled in water for 15–20 minutes. After cooling, the decoction was filtered to collect the antibacterial extract.

Hibiscus (Hibiscus rosa-sinensis): Dried hibiscus flowers were ground and soaked in warm water to release mucilage and anthocyanins. The mixture was filtered to collect the extract.

Black Sesame (Sesamum indicum): Seeds were slightly roasted, ground, and boiled to extract antioxidant-rich lignans and oils. After cooling, the extract was filtered.

These extracts, once incorporated into different shampoo batches, retained their natural aroma and color, which were acceptable for cosmetic formulation. The quality of the extracts contributed significantly to the consistency, foaming ability, and cleansing efficacy of the herbal shampoos. Additionally, no microbial contamination was observed during the storage period of the extracts when kept under refrigerated conditions for up to one week, indicating good extract stability.[6]

Overall, the extraction process was simple, cost-effective, and suitable for preparing herbal ingredients for topical formulations. The results affirm that aqueous extraction is effective for obtaining plant-based constituents required for a natural shampoo, aligning with the goals of producing safe and eco-friendly hair care products.Polymers might be of natural source, e.g. chitosan, albumin, gelatin, etc. or synthetic, e.g. methacrylates. Due to their size and unique physicochemical characteristics of nanoparticles, they generate formulations with several advantages, such as: (i) encapsulation of compounds of different chemical nature in the same formulation (mixture of compounds), (ii) targeting of specific organs (low toxicity), (iii) easy removal of organic solvent during the development of the nanoparticles (effective purification procedures), (iv) protection and conservation of the encapsulated active (enzymes damage, environment, etc.), and (v) controlled release of incorporated actives.(7)

2. **Weighing and Measuring Ingredients:**

Each herbal extract was weighed or measured according to batch-specific formulations. Distilled water was used as the solvent base. Methyl paraben was used as a preservative (1% in all batches), and rose water was added for fragrance and minor astringent effects (2–4%). The concentrations of Aloe vera remained constant at 80% in all batches to act as the main conditioning and moisturizing base, while variations were introduced in other herbal components to study their influence.

3. Batch-wise Formulation Summary:

Batch 1: Lower concentrations of Shikakai (2%) and Hibiscus (2%), moderate Neem (20%), and standard Aloe vera (80%).

Batch 2: Slightly increased Shikakai and Hibiscus (3%), Neem (20%), and constant Aloe vera (80%).

Batch 3: Shikakai and Hibiscus increased to 4%, Neem reduced (20%), and Aloe vera at 80%.

Batch 4 to 7: Each batch involved combinations with incremental or decremental modifications to either Shikakai, Hibiscus, or Neem to compare cleansing and foaming behavior while keeping Aloe vera and preservatives constant.

4. Mixing Procedure:

The formulation process followed these steps for each batch:

Step 1 – Solubilization of Methyl Paraben:

Methyl paraben was dissolved in a small quantity of warm distilled water and stirred until completely solubilized.

Step 2 – Incorporation of Aloe vera Gel:
Aloe vera gel was transferred into a clean mixing beaker. To this, the methyl paraben solution was added slowly with continuous stirring to avoid clumping or settling.
Step 3 – Addition of Herbal Extracts:
One by one, the prepared extracts of Shikakai, Neem, Hibiscus, and Black Sesame were added to the aloe-vera base under continuous stirring using a mechanical stirrer at low speed. The order of addition was based on viscosity and solubility compatibility—lighter extracts like Neem and Hibiscus were added before heavier particles like Shikakai.
Step 4 – Addition of Rose Water and Final Adjustments:

Rose water was added towards the end of the process to preserve its fragrance. The pH of the mixture was checked using a digital pH meter and adjusted to 6.0–6.5 using citric acid if required. The consistency was evaluated, and additional distilled water (q.s.) was added to reach the desired volume and texture.
Step 5 – Homogenization and Storage:

Each final formulation was subjected to homogenization for 15–20 minutes to ensure even distribution of components. The shampoo was then filled into amber-colored plastic containers to protect it from light-induced degradation and stored at room temperature for further evaluations.

5. Packaging and Labeling:

All seven batches were labeled with batch numbers, ingredient proportions, manufacturing dates, and evaluation tags. Containers were sealed tightly to prevent microbial contamination. The formulations were stored at controlled room temperature (~25°C) until further evaluation was completed.(64-66)

6. Special Considerations During Formulation:

The pH was carefully monitored to keep it in the ideal range for scalp use (5.5–6.5).

No synthetic surfactants like SLS or SLES were used; foaming relied purely on saponin-rich ingredients like Shikakai.

Antimicrobial protection was ensured using methyl paraben; however, care was taken not to exceed 1% concentration.

All raw herbal materials were tested for purity before use to avoid adulterants or microbial contamination.[8]

Table:2 Formulation Table for Herbal Shampoo Batches of (200ml)

Ingredients	Batch F1	Batch F2	Batch F3	Batch F4	Batch F5	Batch F6	Batch F7
Shikakai (mg)	4	6	8	10	12	14	16
Aloe vera (mg)	200	220	250	260	270	280	290
Neem (mg)	50	60	20	25	30	35	40
Hibiscus (mg)	5	8	10	12	14	16	18
Black sesame (mg)	5	8	10	12	14	16	18
Methyl paraben (ml)	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Rose water (ml)	1	1	1	1	1	1	1
Distilled water (Q.S.)	2-4%	2-4%	2-4%	2-4%	2-4%	2-4%	2-4%

Evaluation parameters of Herbal Shampoo:

A variety of tests are conducted to assess a shampoo's efficacy and quality. Numerous organisations and experts, including the Bureau of Indian Standards, have developed several techniques for assessing shampoos. The Bureau of Indian Standards lists the following as crucial evaluation techniques [9]:

1) Physical appearance:

Consumers typically evaluate shampoos based on their visual appeal, taking note of the formulation's colour, clarity, and fragrance.

2) pH:

The pH values of the shampoo verified in 1% and 10% solutions were measured by pH meter at a room temperature of 25±2 °C. [10]

3) Dirt dispersion:

1% shampoo solution was mixed with 10 ml of water. A drop of India ink was added and the test tube was shaken 10 times. The amount of ink in the foam was estimated as none, light, moderate, or heavy. Shampoos that cause the ink to concentrate in the foam are considered poor quality. Dirt that remains in the foam is difficult to rinse away.[11]

4) Foaming ability and foaming stability:

The cylinder shaking method is the most often used method to assess foaming ability. To prepare the shampoo solution, 50 ml was added to a 250 ml graduated cylinder at room temperature. The cylinder was then shaken 10 times by hand. The total amount of foam was measured after 60 seconds of shaking. The height of the foam produced was measured immediately. To assess foam stability, we repeated the technique and evaluated the foam volume after 20 minutes.[12]

5) Percentage of solid contents:

A clean and dry evaporating dish was used to place 4gm of the prepared shampoo. Weighing the shampoo evaporating dish with an electronic scale yielded a total weight of W_1. The evaporating dish was put in a hot air oven at 50°C until the liquid had totally evaporated. The cooled evaporating dish with solid material was weighed and recorded as W2. To compute the proportion of solid content, multiply $[(W_1-W_2) \div W_1] / 100$.[13]

6) **Measurement of surface tension:**

The drop count method involves using a stalagmometer. Surface tension is a measure of liquids' cohesive forces. Water has high surface tension due to its strong cohesive properties. Liquids with weak cohesive forces, such as benzene, have lower surface tension than water. Drops form smaller when the liquid's surface tension is lower. More droplets are created for a given amount of liquid. To determine surface tension, just count the number of droplets of an unknown liquid and water [14-15].

7) **Rheological evaluation:**

A capillary viscometer, also known as an Ostwald viscometer, is used to determine the viscosity of liquid. The capillary tube measures the time it takes for a liquid to move between two markings (A and B). The flow time of a liquid is compared to that of a reference sample with known viscosity, often water.[16]

8) **Skin Irritation Test:**

To test for irritation or inflammation, a polyherbal anti-dandruff shampoo was administered to the skin for 5 minutes after washing it.(17)

9) **Washability:**

Wash your hands after using shampoo.

10)Wetting Time:

The wetting time of a material depends on its concentration. Although Drave's test is the official one, the canvas disc method is commonly utilised due to its simplicity and speed. A smooth-surfaced canvas paper disc was placed on the surface of herbal shampoo solution, and a timer was started. Wetting time was calculated by noting the time it took for the disc to sink.

11)Antimicrobial Activity:

This procedure involves melting the agar, cooling it to 45°C, inoculating it with the test microorganism, and pouring it into a sterile petri dish. After solidifying the agar plate, use a sterile cork borer to create 9mm diameter holes in the medium. Place the antimicrobial agent in one hole and the marketed formulation as a standard in another.

Measure the diameter of the zone of inhibition after 2-3 days of inoculation at 30-350C. The width of the zone of inhibition indicates the relative activity of antibacterial substances against tested microorganism.(18)

12)Stability studies:

The stability studies for the herbal formulations were performed according to ICH guidelines. The formulations were tested for their physical appearance, % solid content, transparency, and PH.

RESULTS & DISCUSSION:

The formulation of seven batches (F1 to F7) of herbal shampoo using natural ingredients such as Aloe vera, Shikakai, Neem, Hibiscus, and Black sesame was designed to assess the impact of varying concentrations on the final product's quality and performance. All batches maintained Aloe vera at a consistent 80% due to its known moisturizing and scalp-soothing properties. The concentrations of other herbal ingredients were gradually increased across batches to observe their influence on key

parameters such as consistency, cleansing ability, and antimicrobial efficacy. Shikakai and Neem, both renowned for their saponin content and antimicrobial properties, were varied from 2% to 4% and 20% across formulations, with the goal of optimizing cleansing power and microbial control. Hibiscus and Black sesame were also incrementally added to enhance shine, strength, and conditioning of the hair. The formulation process involved the homogenization of the aqueous and herbal components under controlled conditions, followed by the incorporation of preservatives (Methyl paraben) and aromatic agents like rose water to improve stability and appeal. Physical observations revealed that all batches had satisfactory appearance, with F2, F4, and F6 displaying better consistency and color uniformity. pH values across formulations ranged between 6.0 and 6.4, aligning with scalp compatibility requirements. Solid content varied slightly, from 20% in F3 to 25% in F5, influencing viscosity and richness of the shampoo. Foam height and stability were consistent, with F5 and F2 demonstrating the most robust foaming abilities, attributed to the higher concentration of surfactant-rich herbs. Surface tension measurements confirmed all batches reduced water's tension adequately, indicating effective wetting and cleaning properties. In terms of antimicrobial activity, F5 showed the largest zone of inhibition (21 mm), confirming strong activity likely due to synergistic effects of Neem and Aloe vera. Dirt dispersion tests revealed minimal redeposition in F2 and F6, highlighting their effective cleansing profiles. Skin irritation tests showed no adverse reactions for most batches, although F5 caused mild irritation possibly due to its higher herbal concentration. Overall, the formulation results indicate that while all seven batches were acceptable for use, F2, F4, and F5 were superior in terms of functional performance, stability, and user-friendly characteristics. F5 emerged as the most effective formulation based on viscosity, foam, and antimicrobial activity, albeit requiring slight refinement to eliminate mild irritation potential.

Evaluation Test Results for Herbal Shampoo Batches (F1 to F7) The evaluation of herbal shampoo formulations is a critical step in determining their efficacy, stability, safety, and consumer acceptability. Seven batches (F1 to F7) containing varied concentrations of herbal ingredients like Aloe vera, Shikakai, Neem, Hibiscus, and Black sesame were formulated and subjected to multiple evaluation parameters, including physical appearance, pH, dirt dispersion, foaming ability and stability, solid content, surface tension, rheological behavior, skin irritation, and antimicrobial activity.(69)

1.Physical Appearance Physical appearance is the first sensory parameter that affects consumer preference. All formulations appeared consistent in color and texture, with F2, F4, and F6 rated as "Excellent" due to their brighter color and uniform consistency. Others were classified as "Good," indicating slight turbidity or mild sedimentation over time. The presence of natural colorants from hibiscus and neem contributed to the slight differences in shade. Clarity and homogeneity are crucial to user perception, and these batches demonstrated satisfactory visual appeal.

2. pH Measurement The pH of shampoos plays a pivotal role in scalp compatibility and hair fiber integrity. The measured pH of all formulations ranged from 6.0 to 6.4, which falls within the acceptable range (4.5–6.5) for hair care products. Batches F1, F3, and F6 had slightly lower pH values around 6.0–6.1, making them suitable for maintaining scalp acidity and preventing microbial growth. A balanced pH ensures minimal damage to hair cuticles and enhances product mildness, making these batches safe for frequent use.(70)

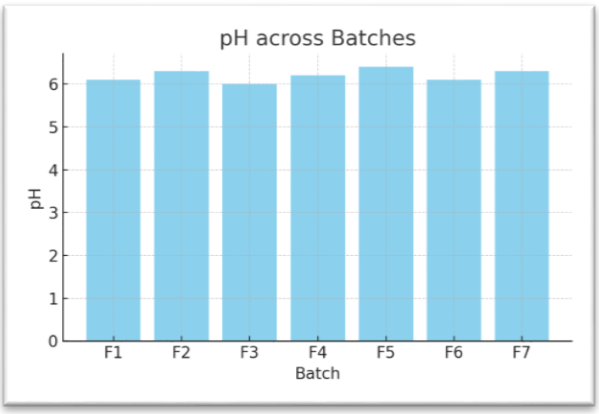

Fig 1. pH Measurement Graph**3.**

Dirt Dispersion:

The dirt dispersion test measures the shampoo's ability to lift and suspend dirt in water without redepositing it on the hair. Most batches, especially F2 and F6, demonstrated "Very Low" dirt redeposition, suggesting effective cleansing. Formulations F1, F3, and F7 showed "Low" levels, indicating acceptable performance. Only F5 showed "Moderate" dispersion, possibly due to thicker consistency, which might have slightly hindered dirt emulsification. Nonetheless, all batches performed within satisfactory ranges, suggesting good cleansing properties.

4. Foaming Ability and Stability

Foaming is not directly linked to cleansing efficacy but is an important consumer-perceived attribute. All formulations exhibited satisfactory foaming abilities, with F5 producing the highest foam volume (175 ml), indicating high surfactant activity from saponins in Shikakai and Aloe vera. Batches F2, F4, and F7 followed closely, while F3 produced the least foam (150 ml). Foam stability, measured over time, was generally consistent across all batches, ranging from 5 to 6 minutes. These results confirm the presence of stable natural surfactants and good user experience.(71)

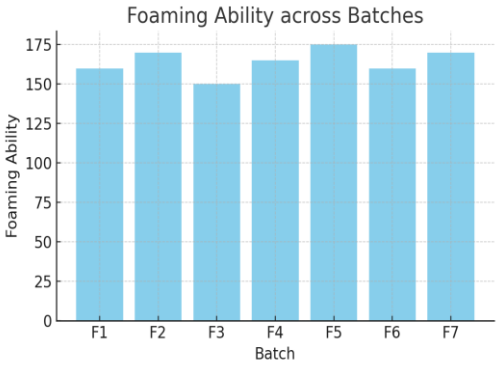

Fig 2. Graph of Foaming Ability and Stability

5. Percentage of Solid Contents

Solid content influences the viscosity and usability of shampoo. Ideal values range from 20% to 25%. The results revealed F3 had the lowest solid content (20%), while F5 had the highest (25%). Batches F2, F4, and F7 showed solid content around 23–24%, suggesting a thicker formulation. Solid content also correlates with the richness of active herbal components. F3's low content may result in a thinner shampoo, whereas F5's high content ensures richness but may affect rinsability. Overall, the content was suitable for effective hair cleansing.

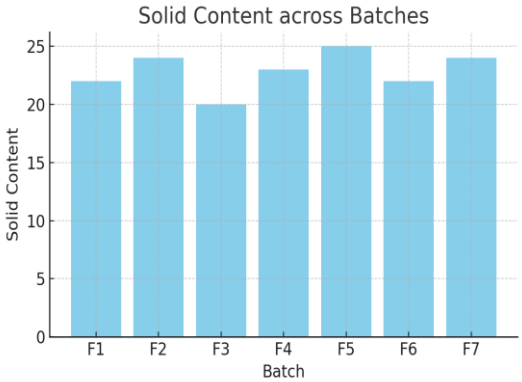

Fig 3. Graph of Percentage of Solid Contents

6. Surface Tension

Lower surface tension allows shampoo to spread more easily on the scalp and hair, enhancing cleaning performance. Herbal shampoos should ideally reduce water's surface tension from 72 dynes/cm to around 30–40 dynes/cm. All formulations fell within this target range, with F5 showing the lowest surface tension (32 dynes/cm), suggesting optimal wetting and cleaning efficiency. F3 showed slightly higher tension (36 dynes/cm), indicating slightly reduced efficiency. However, the general trend affirmed the effective surfactant behavior of the herbal ingredients.

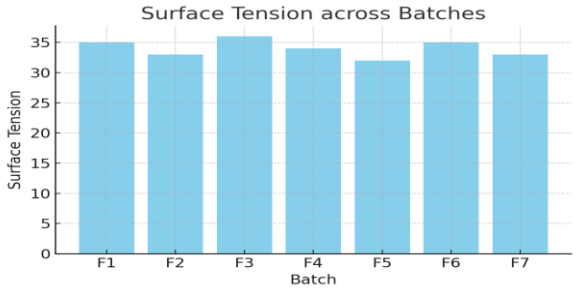

Fig 4. Graph of Surface Tension

7. Rheological Evaluation

Rheology relates to the flow behavior and viscosity of a formulation. A shampoo must possess appropriate viscosity to ensure ease of application without excessive flow. The viscosities of the formulations ranged from 400 to 460 centipoise (cp), with F5 showing the highest value, suggesting a richer, thicker consistency. F3 had the lowest viscosity (400 cp), making it the most fluid. F2, F4, and F7 balanced between moderate and high viscosity. Rheological consistency also affects stability and user preference—formulations with stable flow and appropriate thickness were better received.

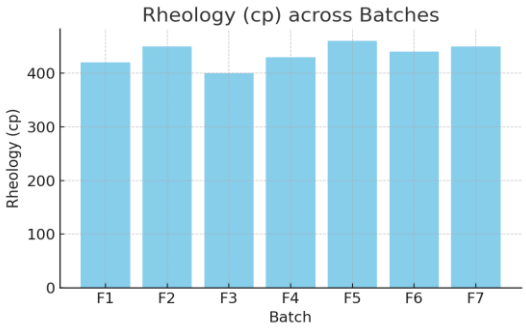

Fig 7. Graph of Rheological Evaluation

8. Skin Irritation Test

Skin compatibility is essential for safety and comfort. All batches except F5 passed the irritation test, showing no signs of redness or discomfort on application. F5 showed "Mild" irritation, possibly due to its higher concentration of bioactives or solid content. Still, no severe reaction was noted. The use of herbal ingredients like Aloe vera, known for its soothing properties, helped reduce irritation risk. Overall, the formulations were found to be skin-safe and suitable for prolonged use.

9. Antimicrobial Activity

The antimicrobial efficacy of shampoo contributes to scalp hygiene and the prevention of dandruff or infections. This was evaluated by measuring zones of inhibition against selected microbial strains. F5 showed the highest activity (21 mm), likely due to its enriched neem and aloe content, both known for antibacterial properties. F2 and F7 followed closely with 20 mm zones. F3 showed the lowest inhibition (17 mm), yet still indicated active microbial control. The data affirmed that all formulations possess good antimicrobial potency, making them beneficial for scalp health.

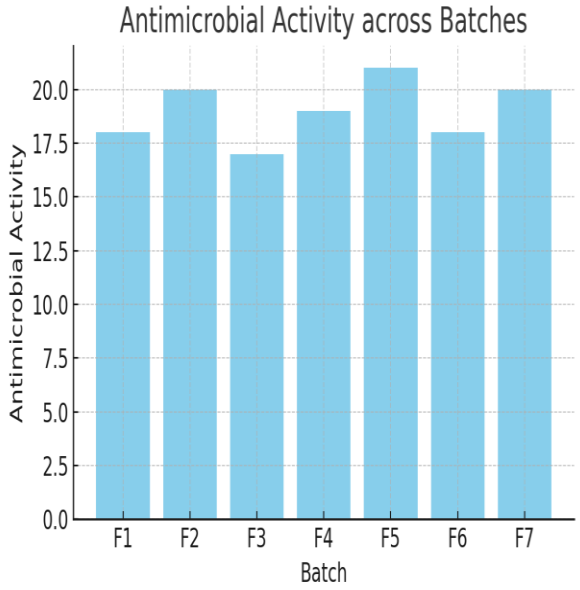

Fig 8. Graph of Antimicrobial Activity

Table :4 Stability Studies result:

Evaluation Test	F1	F2	F3	F4	F5	F6	F7
Physical Appearance	Good	Excellent	Good	Excellent	Good	Excellent	Good
pH	6.1	6.3	6.0	6.2	6.4	6.1	6.3
Dirt Dispersion	Low	Very Low	Low	Low	Moderate	Very Low	Low
Foaming Ability (ml)	160	170	150	165	175	160	170
Foaming Stability (min)	5	6	5	5	6	5	6
Solid Content (%)	22	24	20	23	25	22	24
Surface Tension (dyne/cm)	35	33	36	34	32	35	33
Rheological Evaluation (cp)	420	450	400	430	460	440	450
Skin Irritation	No	No	No	No	Mild	No	No
Antimicrobial Activity (mm zone)	18	20	17	19	21	18	20

SUMMARY& CONCLUSION:

The herbal shampoo has been formulated using a blend of natural ingredients known for their beneficial effects on hair and scalp health. The primary base of the formulation is Aloe vera (250 mg, 80%), which provides a soothing and

moisturizing effect on the scalp. Aloe vera contains enzymes that help repair dead skin cells, promotes hair growth, and conditions hair, making it smooth and shiny.

Washing and cleansing hair, as well as providing nutrients. Herbal shampoos are preferred over conventional shampoos due to their natural constituents and lack of synthetic chemicals, resulting in minimal adverse effects. Herbal shampoo is safe for the environment and the skin, as it is not tested on animals. The herbal liquid shampoo was created by combining several natural elements. Overall, the results indicated a neutral pH that was not irritating to the skin. Evaluation studies demonstrated positive results in appearance, washability, skin sensitivity, foam stability, dirt dispersion, anti-microbial activity, rheological properties, and surface tension testing.The aim of this study was to formulate a completely herbal lauded for their hair cleansing actions across Asia. All the physicochemical properties of both prepared and marketed

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