



## FORMULATION AND EVALUATION OF HIBISCUS MEDICATED ANTIMICROBIAL, ANTIFUNGAL, AND CONDITIONING GEL SHAMPOO.

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**ABSTRACT** **Background:** The leaves and flowers of *Hibiscus rosa-sinensis* have hair growth-promoting and anti-greying properties as well as excellent antimicrobial and antifungal activity. Active ingredients when added in shampoos which classify them as drugs. Medicated shampoos, in addition to cleansing and conditioning, deliver extra benefits to the hair and scalp.

**Objectives:** The objective of this study was to formulate a shampoo making use of the therapeutic activities of *Hibiscus rosa-sinensis* Linn.

**Methods:** The study was carried out using standardized procedures at ST. WILFRED'S INSTITUTE OF PHARMACY, Panvel from 21 August 2021 to 21 December 2021. Our study included identification, quantitative analysis, extraction, pre-formulation studies, evaluation along with all other standard procedures required.

**Results:** The alcoholic extracts of *Hibiscus rosa-sinensis* Linn. showed antimicrobial and antifungal activity. The formulated shampoo preparation passed all the recommended standard evaluation tests viz: Organoleptic properties, rheological evaluations, pH, percent solid test, foam producing, and foam retention capability, wetting time, surface tension, dirt dispersion, and cleansing action.

**Conclusion:** The formulated shampoo revealed the ideal characteristics of the shampoo and is devoid of any harmful preservatives like paraben. It can be used as a safer alternative to its synthetic counterparts.

**KEYWORDS :** Hibiscus, Shampoo, Antimicrobial, Antifungal, Formulation, Evaluation.

### INTRODUCTION:

Cosmetics and personal care products have played an essential role in our lives for thousands of years. Today, an increasing number of products are aimed at specific target audiences and gain importance in groups that have not received as much attention in the past. Innovation, creative thinking, and problem-solving are essential skills needed in the cosmetic industry. However, the most important skill is a strong foundation and basic understanding of the main principles. Cosmetic science is a delicate blend of a variety of knowledge, including chemistry, biology, formulation science, pharmacology, marketing, and law<sup>1</sup>.

Shampoos remove sebum, sweat, environmental dirt, beautify hair and make it easy to handle. From their chemical nature, today, shampoos are surfactant-based preparations. Therefore, their cleaning principle is emulsification. Surfactants surround and trap tiny droplets of fat, which in this form are rinsed from the hair and scalp. Insoluble, particulate soil is eliminated by electrostatic repulsion between the dirt and the hair fiber assisted by repulsion between the surfactant molecules adsorbed onto the hair fiber and those dissolved in the soil. Most commonly, shampoos are colloidal dispersions of various surfactants in water. The fundamental components of a classical shampoo are cleansing agents, thickeners, and water. Usually, additives are incorporated in the formulations to help the cleansing process, enhance the aesthetic properties, increase foaming, and make the hair shine. Active ingredients when added in shampoos which classify them as drugs. Medicated shampoos, in addition to cleansing and conditioning, deliver extra benefits to the hair and scalp<sup>1</sup>.

*Hibiscus rosa-sinensis* Linn. Family: Malvaceae, is a glabrous shrub widely cultivated in tropical Asia as an ornamental plant and has several forms with varying colors of flowers. In medicine, the red-flowered variety is preferred<sup>2</sup>. The extracts of leaves and flowers are promoters of hair growth<sup>3</sup>. According to traditional texts, the leaves and flowers of *Hibiscus rosa-sinensis* have hair growth-promoting and anti-greying properties<sup>2</sup>. Alcoholic extracts of *Hibiscus rosa-sinensis* Linn. exhibit antimicrobial and antifungal activities. These facts and findings from various literature and our research encouraged us to formulate a shampoo that utilizes the therapeutic benefits of concentrated extract of *Hibiscus rosa-sinensis* Linn.

### Materials and Methods:

#### Procurement of samples:

The matured flowers and leaves of *Hibiscus rosa-sinensis* Linn. were

procured from the Dadar flower market and were verified by local farmers. The samples were subjected to cleaning, sorting & grading operations and then utilized for further processing.

### Chemicals:

All chemicals and materials for the present study were collected in optimal form from the SRL chemicals and Astron chemicals. All the chemicals used were of analytical grade.

### Extraction:

The solvent extraction was carried out using standard procedures. 20g of fresh, dried leaves and petals of *Hibiscus rosa-sinensis* Linn. were crushed to powder form and extracted separately using the Soxhlet Extraction method for 24 hours at 60°C using 500ml of Ethanol as a solvent. The extracted solvent was further concentrated in a water bath to obtain solid extract and stored in vials for subsequent uses.<sup>4</sup>

### Antimicrobial and Antifungal screening:

Bacteria seeded plate: Well method was performed to test the antibacterial and antifungal activities of the dried ethanolic extracts of *Hibiscus rosa-sinensis* Linn. against gram-positive bacteria, gram-negative bacteria, and fungi. Fluconazole injection USP, Ceftriaxone injection, and Gentamicin injection were used as standard drugs. The sensitivity of the micro-organisms was measured by calculating the zone of inhibition and comparing it with the zone of inhibition of the standard.

### The results obtained are as follows:

Sample Name	Name of Organism		
	<i>E. coli</i> (2.9 x 10 <sup>6</sup> CFU/ml)	<i>S. aureus</i> (1.5x 10 <sup>6</sup> CFU/ml)	<i>C. Albicans</i> (2.4 x 10 <sup>6</sup> CFU/ml)
	<b>Zone of inhibition (mm in diameter)</b>		
<b>Hibiscus Petal Extract (Ethanol)</b>	21.27	20.87	16.80
<b>Hibiscus Leaves Extract (Ethanol)</b>	16.93	15.31	18.24
<b>Fluconazole Injection USP</b>	30.51	29.24	27.12
<b>Ceftriaxone Injection</b>	40.41	38.36	38.09
<b>Gentamicin</b>	33.90	27.08	24.72

**Formulation Process:**

Add SLS to 70 ml of water in a water bath and dissolve completely with continuous stirring. Add Glycerin and Sodium Citrate and dissolve completely. Set the temperature to 60 °C and slowly add HPMC in small quantities with continuous stirring. Add the ethanolic extracts of *Hibiscus rosa-sinensis* along with perfume in sufficient quantities and make up the volume.

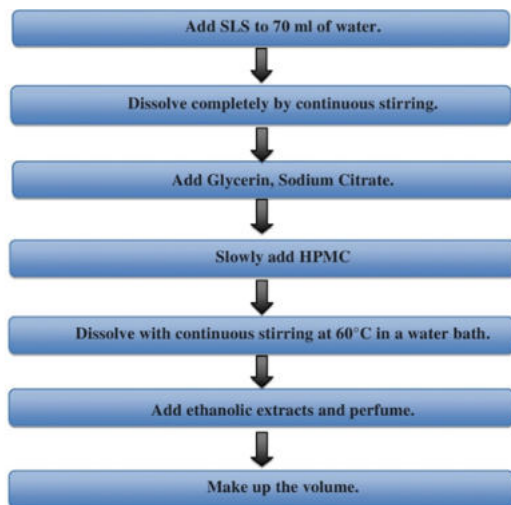


Figure 1: Formulation process.



Figure 2: Formulated Shampoo.

**Formulation Table:**

SR. NO.	INGREDIENTS	QUANTITY	USE
1	SODIUM LAURYL SULPHATE (SLS)	10 g	SURFACTANT
2	SODIUM CITRATE	1.0 g	VISCOSITY ENHANCER
3	HYDROXYPROPYL METHYLCELLULOSE (HPMC) KM4	3.0 g	THICKENING AGENT
4	GLYCERIN	5.0 g	VISCOSITY ENHANCER
5	ORANGE OIL (PERFUME)	Q.S.	FRAGRANCE
6	DISTILLED WATER	100 ml	VEHICLE
7	HIBISCUS EXTRACT (PETALS)	Q.S.	ACTIVE INGREDIENT
8	HIBISCUS EXTRACT (LEAVES)	Q.S.	ACTIVE INGREDIENT

**Evaluation Of Shampoo:**

**Organoleptic evaluation:**

The formulation was evaluated for physical and visual parameters including color, odor, clarity, foaming ability.

**Detergency ability:**

In one percent solution of each shampoo, a soiled hair swatch was washed with 250 ml of one percent solution of each shampoo for 150 seconds in 90 rotate per minute by rotary in 40 °C respectively. It was then dried using a hairdryer and further dried in an oven at 60 °C for 4 h to ensure uniform moisture content and re-weighed. Finally, the

percentage of detergency power was calculated using the following equation: DP is the percentage of detergency power C is the weight of sebum in unwashed swatches, and T is the weight of sebum in the swatches after washing.  $DP = 100(1-T/C)^5$

**Rheological evaluations:**

The viscosity of the shampoo was determined by using Brookfield Viscometer at 26.5°C.

**Determination of pH:**

The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C and the pH was measured with the help of a calibrated pH meter <sup>6</sup>.

**Determine percent of solids contents:**

A clean dry evaporating dish was weighed and 4 gm of shampoo was added to the evaporating dish. The dish and shampoo were weighed. The exact weight of the shampoo was calculated only and put the evaporating dish with shampoo was placed on the hot plate until the liquid portion was evaporated. The weight of the shampoo only (solids) after drying was calculated <sup>7</sup>

**Foaming ability and foam stability:**

The cylinder shake method was used for determining foaming ability. 50 ml of the 1% shampoo solution was put into a 250 ml graduated cylinder, covered with the hand, and shaken 10 times. The total volumes of the foam contents after 1-minute of shaking were recorded. The foam volume was calculated only. Immediately after shaking the volume of foam at 1-minute intervals for 4 minutes were recorded <sup>8</sup>

**Cleaning action:**

5 grams of wool yarn were placed in grease, after that, it was placed in 200 ml. of water containing 1 gram of shampoo in a flask. The temperature of the water was maintained at 35°C. The flask was shaken for 4 minutes at the rate of 50 times a minute. The solution was removed and the sample was taken out, dried, and weighed. The amount of grease removed was calculated by using the following equation:  $DP = 100(1-T/C)$  In which, DP is the percentage of detergency power, C is the weight of sebum in the control sample and T is the weight of sebum in the test sample <sup>9</sup>.

**Dirt Dispersion:**

Two drops of shampoo were added to a large test tube containing 10 ml of distilled water. 1 drop of India ink was added; the test tube was stoppered and shaken ten times. The amount of ink in the foam was estimated as None, Light, Moderate, or Heavy <sup>6</sup>.

**Surface tension measurement:**

Measurements were carried out with a 10% shampoo dilution in distilled water at room temperature. Thoroughly clean the stalagmometer using chromic acid and purified water. Because surface tension is highly affected by grease or other lubricants. The data is calculated by the following equation:

$$R2 = [(W3 - W1) n1 / (W2 - W1) n2] * R1$$

Where W1 is the weight of the empty beaker. W2 is the weight of the beaker with distilled water. W3 is the weight of the beaker with a shampoo solution. n1 is no. of drops of distilled water. n2 is no. of drops of shampoo solution. R1 is the surface tension of distilled water at room temperature. R2 is the surface tension of the shampoo solution <sup>6</sup>.

**Wetting time:**

The canvas was cut into 1-inch diameter discs having an average weight of 0.44g. The disc was floated on the surface of the shampoo solution 1%w/v and the stopwatch started. The time required for the disc to begin to sink was measured accurately and noted <sup>8</sup>

**Stability studies:**

The thermal stability of the shampoos was studied by placing them in glass tubes in a humidity chamber at 45 °C with 75% relative humidity as well as in a refrigerator at 4 °C and comparing them to the same shampoos kept at a room temperature of 25 °C. The thermal stabilities were observed after zero, four, and eight-week storage periods. Their appearances and physical stabilities were inspected for two months <sup>10</sup>.

**RESULTS AND DISCUSSION:**

Sr. no.	Evaluation Tests	Results Obtained
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1	COLOUR	LIGHT BROWN
2	ODOUR	FRAGRANT
3	CLARITY	CLEAR, TRANSPARENT
4	FOAM PRODUCING ABILITY	EXCELLENT
5	DETERGENCY ABILITY	85%
6	VISCOSITY	10000 mPa.s
7	Ph	6.5
8	% SOLID CONTENTS	20% W/V
9	FOAMING ABILITY AND FOAM RETENTION	CONSTANT AT 180 ml
10	CLEANSING ACTION	GOOD
11	DIRT DISPERSION	NONE
12	SURFACE TENSION	32.04 dynes/cm
13	WETTING TIME	2.5 sec
14	STABILITY STUDIES	GOOD

#### Physical and visual evaluation:

The formulated shampoo has an appealing physical appearance with a pleasing fragrance and excellent foaming.

#### Detergency ability:

Although cleaning of soil/sebum removal is the primary aim of a shampoo, experimental detergency evaluation has been difficult to standardize, as there is no real agreement on a standard soil, a reproducible soiling process, or the amount of soil a shampoo should ideally remove.

#### Rheological evaluations:

At low rpm, shampoo showed high viscosity and increase in the shear rate the viscosity of the shampoos drops, this is a favorable property that eases the spreading of the shampoos on hair.

#### pH:

The pH of shampoo was found to be 6.5 which complies with the standard specifications. The pH of shampoos is important for improving and enhancing the qualities of hair, minimizing irritation to the eyes, and stabilizing the ecological balance of the scalp. The pH is one of the ways to minimize damage to the hair. Mild acidity prevents swelling and promotes tightening of the scales, thereby inducing shine.

#### Percent Solid Contents:

If the shampoo has too many solids it will be hard to work into the hair or too hard to wash out. The percent of solid content was found to be 20% as a result it was easy to wash out.

#### Foaming and Foam retention:

Although foam generation has little to do with the cleansing ability of shampoos, it is of paramount importance to the consumer and is, therefore, an important criterion in evaluating shampoo. The foam showed an initial volume of 190ml which declined to 185ml and stayed constant at 180ml at 1-minute intervals. Therefore, the formulated shampoo showed excellent foaming and foam retention capabilities.

#### Cleaning action:

Cleaning action was tested on wool yarn in grease. Although cleaning of soil/sebum removal is the primary aim of a shampoo, experimental detergency evaluation has been difficult to standardize, as there is no real agreement on a standard soil, a reproducible soiling process, or the amount of soil a shampoo should ideally remove.

#### Dirt Dispersion:

Shampoo that causes the ink to concentrate in the foam is considered poor quality, the dirt should stay in the water. The ink that stays in the foam will be difficult to rinse away. It will redeposit on the hair. As the results indicated, no ink was retained in the foam. Therefore, the prepared formulation is satisfactory.

#### Surface Tension:

A proper shampoo should be able to decrease the surface tension of pure water to about 40 dynes/cm. Surface tension reduction is one of the mechanisms implicated in detergency<sup>6</sup>. The reduction in surface tension of water from 72.8 dynes/cm to 32.04 dynes/cm by the herbal shampoos is an indication of their good detergent action.

#### Wetting Time:

Wetting efficiency is considered to be higher if the disc takes less time for sinking. The formulated shampoo showed a good wetting time of only 2.5 sec.

#### Stability studies:

The stability and acceptability of organoleptic properties (odor and color) of formulations during the storage period indicated that they are chemically and physically stable.

#### CONCLUSION:

The present study shows that the ethanolic extracts of traditionally used shrub *Hibiscus rosa-sinensis* Linn. showed excellent antimicrobial and antifungal activities along with other beneficial characteristics. Herbal-based shampoos are more effective in terms of safety and ease of manufacturing and from an economic point of view. The awareness and need for cosmetics with herbal ingredients are on the rise, as it is strongly believed that these products are safe and free from side effects. The formulated shampoo preparation shows the ideal characteristics of shampoo and is free from harmful preservatives like paraben.

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