

**OPTIMIZATION AND PERFORMANCE ASSESSMENT OF HERBAL
HAIR COLORING AGENT BY USING BERLERIA PRIONITIS****¹MD. Afreen Begum, ²G. Amulya, ³M. Srujan, ⁴Dr. G. Tulja Rani**

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ABSTRACT

Most of the people are facing the problem of white/grey hair in this modern era and the reasons are many including the reduced synthesis of melanin as it is the main cause of hair colour which is synthesised by melanocytes. Other factors include age, vitamin b12 deficiency, stress, Genetics, Autoimmune diseases etc. So in these conditions hair dye plays an important role. As herbal products are in high demand, herbal dyes are showing a lot of potential over synthetic dyes in pharmaceutical, cosmetics, textile and food industries. In this study, an effort has been made to formulate and standardize herbal hair dye for ensuring quality, stability and efficacy. This dye was subjected to several tests such as organoleptic properties, physicochemical profile, phytochemical analysis, rheological

properties, patch test and stability studies. The herbal dye was formulated with natural ingredients following the general principles of formulation. The findings suggested that the formulation had good stability and quality, and was appropriate for use as a hair dye. In conclusion, the herbal hair dye is safe, effective, and natural alternative to chemical dyes and can be developed to meet pharmacopoeial standards. The current study is to develop and assess a polyherbal hair dye from natural plant materials with particular focus on Barleria prionitis flower, which is traditionally used for darkening hair, as an antioxidant and for strengthening hair. An herbal hair dye was prepared with henna, hibiscus flower, amla fruit, beetroot and Barleria prionitis flower powder. The hair dye was subjected to organoleptic, physicochemical, safety, dyeing and stability studies. The addition of Barleria prionitis flower powder was found to enhance the colour intensity, uniformity and hair conditioning in comparison to the dye without the flower. The findings indicate that the herbal hair dye is

safe, effective and has the potential to become a natural alternative to chemical hair dyes.

KEYWORDS: Hair pigmentation, Natural hair coloring, Herbal cosmetics, Herbal hair dye.

INTRODUCTION

The procedures of hair dyeing and hair coloring are widely undertaken to improve appearance, regain original hair color, and conceal grey or white hair resulting from ageing, sun damage or cosmetic treatments. Recently, there is a shift in the use of herbal and natural products because of the awareness of adverse effects of synthetic chemicals. Herbal hair dyes obtained from natural substances are gaining popularity as they not only color the hair but also condition and nourish it without side effects. In ancient times, plants like shikakai, amla, bhringraj, henna and indigo have also been extensively used for enhancing hair quality and providing colour. Herbal dyes are safer, non-toxic, and preserve the integrity of hair, unlike chemical dyes. Given the growing demand for eco-friendly personal care products, the formulation of herbal hair dyes is crucial. So, the current study focuses on the preparation and assessment of a herbal hair dye containing natural components that offer effective hair dyeing, stability and hair care. Along with these well-known herbs, this study focuses on the addition of *Barleria prionitis* flower in the herbal hair dye recipe. *Barleria prionitis* is a medicinal plant with anti-inflammatory, antimicrobial, antioxidant and wound-healing properties. The inclusion of this flower is for improving scalp health, to prevent irritation when applying dye on hair, and to improve hair condition, with improved dye absorption and retention.

HERBAL HAIR DYE

Herbal dye is a natural dye derived from plant sources like roots, leaves, flowers, fruits, seeds, and bark, which is used for safe and environmentally friendly colouration of hair, textiles, cosmetics, food and drugs.

Advantages of Herbal Colouring agents

- Easy to use and safe - derived from plants.
- Non-toxic and non-allergenic.
- Eco-friendly and biodegradable.
- Not harmful for hair and skin.
- Moisturises and nourishes hair.
- No harsh chemicals such as ammonia and peroxide.

- Safe to use over a long period of time.
- Less irritation.

Disadvantages of Herbal Colouring agents

- Fewer colour options than chemical dyes.
- Limited colour options.
- Possibly less colorfastness.
- Time-consuming dyeing process.
- Inconsistent shade.
- Less stable and shorter expiration date.
- Requires mordants for fixation.

AIM AND OBJECTIVE

Aim: The aim of this project is to formulate and evaluate a safe, effective, and stable herbal hair dye using natural ingredients, with a specific emphasis on *Barleria prionitis*. The formulation is intended to provide a healthier, eco-friendly, and sustainable alternative to conventional chemical hair dyes.

OBJECTIVE

- Extract and phytochemical screening of *B. prionitis* leaves.
- Formulate herbal hair colour powder with *B. prionitis*, Henna, Indigo, Amla.
- To evaluate the of *barleria prionitis* flower in enhancing color intensity dye uptake and color stability of the herbal hair
- To study the contribution of *barleria prionitis* in improving hair conditioning and scalp soothing properties of the formulation

INGREDIENTS USED AND THEIR ROLES

Henna is a dye made from the leaves of the *Lawsonia inermis* plant. It's used primarily for hair and skin coloring.

Henna is reddish-brown in color and also provides conditioning to hair.

Uses:

Natural hair coloring Hair conditioner

Used to dye skin (mehndi) Reduces hair loss and dandruff



Fig. No. 01: henna fresh and dried leaves.

2. *Barleria prionitis*

Family: Acanthaceae

Mullu Gorintaku is a medicinal plant used in Ayurveda and folk medicine, particularly for hair, skin and oral health.

Uses

Natural hair dye Strengthens hair roots Reduces hair fall Reduces skin infections Prevents gum bleeding



Fig. No. 2: *Barleria prionitis* fresh and dried leaves.

3. *Hibiscus*

Source: Hibiscus is dried flowers of *Hibiscus rosa-sinensis*

Family: Malvaceae

Uses

Stimulates hair growth, Prevents premature graying, Reduces dandruff, Makes hair soft and shiny, Controls blood pressure, Improves digestion, Acts as an antioxidant, Helps in weight loss



Fig. No. 3: Hibiscus fresh and dried leaves.

4. Amla (Indian Gooseberry)

Amla is the dried or fresh fruit of *Emblica officinalis* (also known as *Phyllanthus emblica*) Family: Euphorbiaceae

Uses

Slows down premature graying, Promote hair growth, Prevent hair fall, Strengthen hair follicles, Darkens and shines hair Improves immunity, Helps in diabetes control, Improves digestion, Acts as a blood purifier, Improves liver function.



Fig No. 4: Amla fresh and dried fruit.

5. Beta vulgaris (Beetroot) Botanical name: Beta vulgaris

Uses

Beetroot is a popular natural dye used in herbal hair colours, cosmetics and food products. It is used to conceal grey hairs, enhance hair luster, and promote healthy hair. It also helps in improving blood circulation, boosting hemoglobin, and improving complexion and health. It is good for the liver, detoxifies and gives energy because of its antioxidant properties. It is also found in herbal lip and cheek tints.



Fig. No. 05: Beeta vulgaris.

Extraction Procedure of Herbal Hair Dye

1. Collection of Plant Material

The first step in obtaining herbal dye is the extraction of the plant part. The portion of the plant with maximum amount of dyeing pigment is dried and used. For instance, leaves are used in henna, flowers in hibiscus and fruits or seeds in beetroot and annatto. The plant part is selected from a pollution-free environment. Healthy, fresh and pest-free plants should be used for quality dye. Once the plant material is collected, it is washed thoroughly with fresh water to remove any dirt, dust, insects or other contaminants. This is necessary to ensure that the dye extraction is not interfered with any contaminants.

2. Drying of Plant Material

The next step is to dry the plant material. This ensures the prevention of spoilage and facilitates pigment extraction. The plant material is dried in thin layers in the shade at ambient temperature. It's important to dry in the shade as sunlight may destroy heat sensitive pigments like anthocyanins and flavonoids. Drying the material can take a few days depending on the thickness of the layers and moisture content of the raw material. Hot air ovens or tray dryers can also be used to accelerate the drying by maintaining a temperature of 40-50°C. However, high temperatures should be avoided as this can result in the loss of volatile and thermolabile components.

3. Size Reduction

After the plant material is dried, it is size reduced. The dried plant material is crushed into a coarse or fine powder using a grinder or mortar and pestle. This process enhances the contact between the solvent and the plant material by increasing the surface area of the plant and thus leads to efficient extraction of the pigments. The powder is kept in a dry and clean container until it is ready for use.

4. Selection of Solvent

Selecting the right solvent is essential in the extraction of herbal dyes. It should dissolve the colours' pigments well, and be safe, non-toxic and cost-effective. Water, ethanol, methanol, acetone, or a combination of water and alcohol are often used as solvents. Water is the most common solvent, as it is safe, cheap and environmentally friendly. But alcoholic solvents are more suitable for extraction of some pigments. Anthocyanins and flavonoids are soluble in water and ethanol, but some carotenoids are better extracted in organic solvents. For most herbal dye preparation used in pharmaceutical and cosmetic products, a water or hydro-

alcoholic solvent is ideal.

5. Maceration Method

Maceration is one of the simplest methods of extracting herbal dyes and is the most widely used technique. A known weight of powdered plant material is used in this procedure and enough amount of solvent is added to this material to make it completely covered. The mixture is stirred and allowed to rest at room temperature over a certain duration of time typically 24-72 hrs. Within this time frame, the solvent enters the plant tissues where the coloring pigments are dissolved. The mixture is at times stirred or shaken to enhance extraction. The mixture is filtered after the necessary time on a muslin cloth or filter paper to filter the liquid extract and the solid residue. The extract of the herbal dye is found in the filtrate.

6. Decoction Method

Another fairly common technique is known as decoction and it is utilized mostly in hard plant materials like roots, bark, and seeds. Here, the plant material is heated in water by boiling the powdered or chopped plant material to a certain duration, normally 30 to 60 minutes. The process of boiling is used to loosen the cell walls, and to free the pigments into the solvent. The mixture is boiled and allowed to cool then filtered. The dye extract is the filtrate. Decoction is also effective in the extraction of tannins, water-soluble pigments.

7. Soxhlet Extraction

Soxhlet extraction is a continuous extraction technique which is applied when a high dye concentration is needed. In this approach, plant material is put in a thimble in a Soxhlet apparatus in a powder form. The solvent is heated and the vapors of this solvent condense and permeate the plant material dissolving the pigments. The pigments extracted are collected in a flask. This process repeats itself until the desired quantity of pigment is extracted. Soxhlet extraction is more effective than maceration or decoction but more expensive and time consuming.

8. Filtration and Concentration

The liquid extract is then filtered after extraction to remove any solid particles left. This filtrate is then concentrated by evaporating the solvent, typically in a water bath or rotary evaporator. This step enhances the concentration of the dye and enhances its stability.

9. Storage of Extract

The extract of the dye is concentrated and kept in clean, airtight and hopefully amber containers to avoid exposure to light, air and water. It is kept in cool temperature to avoid degradation and microbial growth.

Preparation of Herbal Dye

1. Formulation (Powder form)

- Use various powders in a particular proportion to obtain the required color and properties.
- Add the powders into the mixture to form a homogenous mixture. The powder can be then airtight packaged.

2. Formulation (Paste)

- In order to make a paste, add the herbal powder or concentrated extract to warm distilled water until it forms a thick paste.

Additives such as glycerin may enhance consistency and conditioning activity and a preservative (e.g., methylparaben) may enhance shelf life.

- The mixture needs to be neutralized to a safe level (approximately 7) to apply safely to the hair/skin.

Herbal Hair Dye Formulations (F1–F5) Using *Barleria prionitis*

Table No. 01: Table showing the formulation ingredients.

| s.no | Ingredient | Botanical Name | Part Used | F1 (g) | F2 (g) | F3 (g) | F4 (g) | F5 (g) |
|------|---------------------------|-------------------------------|-----------|--------|--------|--------|--------|--------|
| 1 | Henna powder | <i>Lawsonia inermis</i> | Leaves | 70g | 70g | 70g | 70g | 70g |
| 2 | Amla powder | <i>Emblica officinalis</i> | Fruit | 10g | 20g | - | - | - |
| 3 | Hibiscus powder | <i>Hibiscus rosa-sinensis</i> | Flowers | - | - | - | 10g | - |
| 4 | Beetroot powder | <i>Beta vulgaris</i> | Root | 15g | - | 15g | - | 5 |
| 5 | Shikakai powder | <i>Acacia concinna</i> | Pods | - | - | - | - | - |
| 6 | <i>Barleria prionitis</i> | <i>Barleria prionitis</i> | Leaves | 5% | 10% | 15% | 20% | 25% |

2. Preparation (Powder Form) method

The dried plant materials were individually dried in a grinder then sieved through a sieve No. 60 to achieve a homogeneous particle size. Weighed amounts of each ingredient were accurately taken as per the formulation table. Geometric dilution was used to thoroughly mix the powders to come up with a homogeneous mixture. The resulting blend of the powder was kept in airtight and moisture resistant containers to be evaluated further and

used.

3. Herbal Hair Dye Paste (Preparation) (To be used)

To apply to hair, the powder formulation was turned into a paste with distilled water. Paste

Preparation Formula:

Herbal dye powder: 50 g.

Warm distilled water: Adequate amount (say 120-150 mL)

Procedure:

Herbal dye powder (50 g) was put in a clean bowl. Warm distilled water was added gradually and the paste was stirred, until a smooth paste was formed with no lumps. The paste was left to dry after 30 minutes to release the dye. The paste was applied evenly over the grey or white hair, prepared and left to dry to last 60-90 minutes after which the paste was washed off using plain water.



Fig. No. 06: Herbal Hair Dye Paste (Preparation) and application.

EVALUATION TEST FOR HERBAL HAIR DYE

Herbal hair dyes should be scientifically considered to be safe, stable, performing and acceptable to the consumer. Herbal formulations are not as standardized, unlike synthetic dyes because of variations in natural phytochemicals, thus necessitating standardization and testing.

1. Organoleptic Evaluation

Organoleptic evaluation is the sensory evaluation of a herbal hair dye through sight, smell and touch to determine its quality and acceptability. A little of the dye is put on a glass plate in the daytime to test the color, smell, feel and consistency. The formulation is supposed to be of pleasant natural smell, even color and smooth and free-flowing texture. Unpleasant smell could be a sign of microbial growth or decay, and disproportional color could be a sign of

inadequate mixing. Ideally, the dye must be smooth, devoid of lumps, uniform, and pleasantly-smelling.

2. pH Determination

The pH of herbal hair dye is very important, since the normal pH of human scalp lies within the range of 5.5-6.5, 1 g of herbal dye powder is dissolved in 100 ml of distilled water and allowed to stand after 30 minutes, The pH of the supernatant is obtained by using a digital pH meter.

Excessively acidic = scalp irritation.

Excessively alkaline: damage/dryness of hair shafts. Correct pH - protects cuticle and enhances uptake of dyes. The Ideal range of herbal dyes is pH 5.5 -7.0 and is slightly acidic or neutral.

3. Loss on Drying (Moisture Content)

This test identifies the quantity of moisture in the herbal dye powder. The amount of moisture and volatile content in a sample is determined using Loss on Drying (LOD). To begin with, one warms a clean dry weighing bottle or dish in an oven at 100-105 °C and cools it in a desiccator then weighs it (W1). The sample is weighed (W2) and 1-5 g of it is carefully added to the dish. The sample is then put in a hot air oven at 100-105 °C 2-5 hours. The dish is dried and then cooled in a desiccator to prevent the absorption of moisture. The weighed cooled dish is noted to be W3. The drying, cooler and weighing process is repeated until the same weight is achieved. The loss in weight indicates the moisture and volatile matter content in the sample. The percentage loss on drying is calculated using the formula: $LOD (\%) = [(W2 - W3) / (W2 - W1)] \times 100$. This is to ensure that the temperature is taken care of and that the heat-sensitive samples are not overheated. This is also an extensive technique in the pharmaceutical, chemical and food analysis to control quality. Microbial growth, Reduced shelf life, Lump formation, Low moisture are the results of high moisture content.

4. Ash Value

Ash value is a calibration of the total mineral content and impurities, Ash value is a gauge of the total quantity of inorganic material which remains after complete burning of an example, which is utilized in the pharmaceutical and food examination. Clean dry silica crucible is initially heated to approximately 500-600 °C in a muffle furnace and then cooled in a desiccator and weighed (W1). The sample is weighed into the crucible and the mass of the crucible accurately weighed as W2. The sample is then burnt at a slow rate to prevent loss

through spattering and at a higher rate of 500-600C to the point of turning white which is a sign that it is carbon free. The crucible is taken off, allowed to cool in desiccator and weighed as W3. In case the carbon particles remain, then the process is repeated until a constant weight is achieved. The ash is the inorganic salts present or stuck on the sample naturally. The percentage of ash value is calculated using the formula: $\text{Ash (\%)} = [(W3 - W1) / (W2 - W1)] \times 100$. Temperature should be properly controlled so that every ash is burned off. This technique is used to ascertain purity and quality of crude drugs and other substances. Ash is high this means it is contaminated by sand, soil or inorganic matter.

5. Particle Size Analysis

Particle size analysis is the analysis of the distribution of the particle sizes in a sample, usually by sieve analysis. It will be carried out with a series of standard sieves in descending size with the sample on the highest sieve. The sieves are mechanically shaken over a given period of time to separate particles according to size. The content that is left behind on each sieve is taken and weighed so as to determine percentage distribution. This is a common method in pharmaceutical and industrial quality control in order to guarantee uniformity. Particle size influences: Smoothness, Application, Dye penetration, Smaller particles provide a better hair coating.

6. Solubility Test

The herbal dye must be freely soluble in water and this is checked by mixing the powder with some water, and watching it dissolve to form a smooth uniform mixture without any lumps. Assessment of safety is achieved with the help of skin irritation (patch) test where a small portion of dye paste is placed on the inner arm and allowed to stay 24 hours. The area is monitored after the period of exposure to redness, itching, and swelling. In the event that no such reactions are observed, then the herbal dye can be considered safe to use on the skin.

Dyeing Time Period needed to develop hair color. The time of dyeing is the time that it takes a dye to produce the desired color on a material. It is identified by the application of the dye and monitoring the color formation at various times. Adequate dyeing duration will provide effective dyeing without degradation. The strands of hair are immersed in dye paste and monitored after every 10 minutes.

7. Wash Fastness

The capacity of a colored substance to withstand the loss of color upon being washed is

called wash fastness. It is tested by immersing the dyed sample under normal conditions and seeing whether it fades out or leaves a stain on other materials. Good wash fastness means that the dye is resistant to washing off. To determine color retention, hair strands are washed repeatedly.

8. Stability Studies

Stability studies determine the effect of product on quality, color and effectiveness in various environmental conditions over an extended period of time. The sample is kept under different temperatures, humidity and exposure to light over a given duration. It is observed periodically to detect a change in color, odor, texture and performance. Good stability means that the product has not been destroyed due to aging in the shelf life. Formulation is kept under varying temperatures and monitored in terms of Color, Odor, pH, Texture etc.

F1 formulation F2 formulation F3 formulation F4 formulation F5 formulation



Fig. No. 07: Result of Herbal Hair Dye Paste of different formulations after application.

RESULTS AND DISCUSSIONS

Table No. 02: Table showing all the evaluation results of the Formulations (F1–F5)

| s.no | Property | F1 | F2 | F3 | F4 | F5 |
|------|------------------|-------------|-------------------|------------|---------------|-----------------------|
| 1 | colour | light Brown | brown | Dark brown | Dark brown | Deep brown |
| 2 | odour | Mild herbal | Pleasant | Pleasant | Strong herbal | Pleasant & acceptable |
| 3 | Texture | Coarse | Moderately fine | Fine | Smooth | Very fine |
| 4 | pH | 6.8 | 6.6 | 6.4 | 6.2 | 6.1 |
| 5 | LOD | 8.5% | 7.9% | 7.2% | 6.8% | 6.5% |
| 6 | Ash value | 12.4% | 11.8% | 10.5% | 9.5% | 8.9% |
| 7 | Particle size | Coarse | Moderately coarse | Fine | Very fine | Ultra fine |
| 8 | Solubility | Poor | Moderate | Good | Very Good | Excellent |
| 9 | Colour Intensity | Light | Moderate | Good | Very good | Excellent |
| 10 | Coverage | Poor | Fair | Good | Very | uniform and |

| | | | | | | |
|----|--------------------|---------------|-------------|--------|-----------|-----------|
| | | | | | Good | Complete |
| 11 | Stability (Colour) | Slight fading | Mild fading | Stable | Stable | No change |
| 12 | Stability (Odour) | Slight change | Stable | Stable | Stable | Stable |
| 13 | Physical Stability | Poor | Fair | Good | Very good | Excellent |

F5 showed highest stability under storage conditions, confirming optimized formulation.

CONCLUSION

Herbal dyes are extracted in various processes that involve harvesting, drying, reduction of size, choice of solvent, extraction, filtration, concentration and storage. All the steps are significant in the quality and effectiveness of the final dye. Correct extraction will give the herbal dye its natural color, safety and medicinal benefits so it can be used in hair dyes. The current study was able to prepare and test a polyherbal hair dye based on natural plant dyes. Hair color with the use of *Barleria prionitis* flower was much more profound and uniform and conditioning was better than formulations made in the absence of the flower. *Barleria prionitis* was used in various concentrations as a part of the five herbal hair dye formulations (F1-F5) where it was used to formulate the formulation and provide the desired antioxidant activity, shine and hair texture. F1 was a low extract content (5%) and base-dominated formulation and F2 was supplemented with amla to enhance conditioning effects. F3, which contained 15% extract of henna, exhibited potential good coloring and F4 was synergistic on herbs and hibiscus with 20% extract. F5 was determined to be the final optimized formulation, which included a balanced multi-herb system with the maximum concentration of *Barleria prionitis* extract (25%). According to the general analysis, F5 showed better performance with regard to color intensity, stability, and application properties, which suggests that a 25% concentration of *Barleria prionitis* extract is the best to optimize herbal hair coloring formulation.

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