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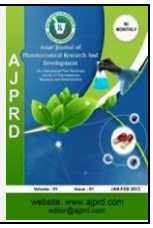

Open Access Research Article

Formulation and Evaluation of Herbal-Based Hair Dye for Cosmetic Applications

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ABSTRACT

Herbal substitutes that are safe, environmentally friendly, and good for hair health are becoming more popular as people become more conscious of the negative consequences of synthetic hair dyes. Using natural ingredients like amla (*Emblica officinalis*), methi (*Trigonellafoenum-graecum*), henna (*Lawsoniainermis*), indigo (*Indigoferatinctoria*), hibiscus (*Hibiscus rosa-sinensis*), bringaraj (*Ecliptaalba*), shikakai (*Acacia concinna*), curry leaves (*Murrayakoenigii*), kalonji (*Nigella sativa*), and beetroot (*Beta vulgaris*), a polyherbal hair dye is the subject of this study. The chosen plant ingredients were dried and ground into a powder using the proper ratios to create the herbal dye. Numerous physicochemical parameters, such as stability, moisture content, pH, organoleptic characteristics, and particle size, were assessed for the produced formulation. The color intensity, dyeing effectiveness, and conditioning effects on hair were also evaluated for marketed henna and natural leaves of henna. The results showed that the marketed henna containing herbal hair dye with beetroot powder (F1) had good stability, conditioning qualities, and coloring capabilities without irritating the scalp or damaging the hair. In addition to providing natural coloring, the substances' bioactive components strengthened, nourished, and encouraged hair development. In conclusion, the created polyherbal hair dye has both cosmetic and medicinal advantages and can be a safe and efficient substitute for synthetic dyes. This study demonstrates the potential of herbal formulations with few adverse effects for use in personal care products.

Keywords: Herbalhairdye, Polyherbal Formulation, Naturalcolorant, Physicochemical Evaluation, Dyeing Efficiency.

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INTRODUCTION

Hair is a crucial aspect of an individual's appearance, significantly influencing their social, cultural, and psychological identities. Hair coloring has been employed since antiquity, utilizing natural substances such as minerals and botanical extracts to alter hair color. The market for hair dyes has markedly expanded in contemporary times due to factors such as aging, fashion trends, and the pursuit of enhanced aesthetic appeal. Nonetheless, most commercially available hair dyes are synthetic and comprise chemicals such as hydrogen peroxide, ammonia, resorcinol, and para-phenylenediamine (PPD), which are associated with various adverse effects, including scalp irritation, allergic dermatitis, hair shaft damage, and potential carcinogenic risks with extended use [1].

In recent years, consumer awareness has increased regarding the safety and environmental implications of synthetic cosmetic goods. This has resulted in heightened interest in herbal cosmetics, regarded as safer, biodegradable, and more compatible with the human body. Herbal formulations employ plant-derived components that fulfill both cosmetic functions and medicinal advantages. In contrast to synthetic colors, herbal hair dyes typically lack harsh ingredients and are less prone to induce toxicity or long-term health risks [2].

Henna (*Lawsoniainermis*) is one of the most extensively utilized and thoroughly researched natural colorants derived from plants. The active ingredient lawsone in henna binds efficiently with keratin in hair, resulting in a reddish-brown hue while simultaneously nourishing the hair. Indigo (*Indigoferatinctoria*) is a significant natural dye that, when combined with henna, yields darker hues from brown to black. These natural colors have been

historically utilized throughout several cultures owing to their safety, accessibility, and efficacy [3]. In addition to colouring agents, several medicinal plants contribute to the overall health and quality of hair. Amla (*Emblica officinalis*) is rich in vitamin C and antioxidants, which help in strengthening hair follicles and preventing premature greying. Bhringraj (*Ecliptaalba*) is known for its role in promoting hair growth and reducing hair fall, while hibiscus (*Hibiscus rosa-sinensis*) acts as a natural conditioner that improves hair texture and shine. Shikakai (*Acaciaconcinna*) is traditionally used asa natural cleanser due to its mild surfactant properties, and fenugreek (*Trigonellafoenum-graecum*) seeds provide proteins and nicotinic acid that nourish the scalp and hair. Other ingredients such as curry leaves (*Murrayakoenigii*), kalonji (*Nigella sativa*), and beetroot (*Beta vulgaris*) contribute additional nutrients, pigments, and bioactive compounds that enhance both the functional and therapeutic properties of the formulation [4].

The efficacy of herbal hair dyes is mostly due to the presence of bioactive phytochemicals, including flavonoids, tannins, alkaloids, and phenolic compounds. These ingredients not only provide natural color but also demonstrate antioxidant, antibacterial, and anti-inflammatory properties, which contribute to scalp health and the prevention of hair-related illnesses (Table 1). Moreover, the utilization of several herbs in a single formulation, known as a polyherbal method, might yield synergistic effects, enhancing the overall efficacy of the product. Polyherbal compositions are extensively recognized in traditional health systems and are progressively being investigated in contemporary cosmetic research thereby improving the overall efficacy of the product [5,6].

Table 1: Biological profile of the selected plants

S. No.	Biological Name	Family	Common Name	Part Used	Major Chemical Constituents	Uses
1	*Emblicaofficinalis*	Phyllanthaceae	Amla	Fruit	Vitamin C, tannins, flavonoids, gallic acid	Strengthenshair, preventsgreying, promotes growth
2	*Lawsoniainermis*	Lythraceae	Henna	Leaves	Lawsone, tannins, flavonoids	Naturaldye, conditions hair, cooling effect
3	*Indigoferatinctoria*	Fabaceae	Indigo	Leaves	Indigotin, indican	Providesdarkcolor,enhancesdyeintensity
4	*Trigonellafoenum-graecum*	Fabaceae	Methi	Seeds	Proteins, saponins, alkaloids, nicotinic acid	Nourishes scalp, reduces dandruff
5	*Hibiscusrosasinensis*	Malvaceae	Hibiscus	Flowers & Leaves	Anthocyanins, flavonoids, mucilage	Conditioner, promotes hair growth
6	*Ecliptaalba*	Asteraceae	Bhringraj	Whole plant	Wedelolactone, flavonoids	Prevents hair loss, promotes growth
7	*Acaciaconcinna*	Fabaceae	Shikakai	Pods	Saponins, tannins	Natural cleanser, maintainshair health
8	*Murrayakoenigii*	Rutaceae	Curry Leaves	Leaves	Alkaloids, antioxidants,vitamins	Prevents greying, strengthens roots
9	*Nigellasativa*	Ranunculaceae	Kalonji	Seeds	Thymoquinone, essential oils	Improves scalp health, reduces hairfall
10	*Betavulgaris*	Amaranthaceae	Beetroot	Root	Betalains, vitamins, minerals	Naturalpigment, enhancescolor

MATERIALS AND METHODS

Materials

All herbal components utilized in the hair dye formulation were either sourced from the local market or processed in the laboratory. Henna powder was procured from a reputable commercial supplier, whereas real henna leaves were gathered, shade-dried at ambient temperature to retain their active compounds, subsequently ground into a fine powder using a mechanical grinder and sieved to achieve uniform particle size. Additional ingredients, including indigo powder, bhringraj powder, hibiscus powder, shikakai powder, methi powder, amla powder, curry leaves powder, kalonji powder, and beetroot powder, were procured in desiccated form from local herbal vendors.

Methods

Preparation of Herbal Hair Dye Formulations

Four distinct formulations (F1, F2, F3, and F4) of herbal hair color were created by altering the ingredient composition, specifically the type of henna (commercial and natural) and the inclusion or exclusion of beetroot powder.

F1: Marketed henna infused with beetroot powder

F2: Marketed henna devoid of beetroot powder

F3: Natural henna with beetroot powder

F4: Natural henna devoid of beetroot powder

All herbal components were meticulously measured in accordance with the formulation specifications (Table 2). The powders were thereafter combined meticulously by geometric dilution to guarantee even dispersion of all constituents. The amalgamated mixture was further triturated to achieve a fine, homogeneous powder appropriate for application. The produced formulations were maintained in hermetically sealed containers at ambient temperature to safeguard them against moisture and environmental contamination until subsequent assessment.

Table 2: Composition of Herbal Hair Dye Formulations

Ingredient (powder form)	F1	F2	F3	F4
Henna	10g	10g	10g	10g
Indigo	3g	5g	3g	5g
Bhringraj	5g	1g	5g	1g
Hibiscus	5g	1g	5g	1g
Shikakai	3g	1g	3g	1g
Methi	2g	1g	2g	1g
Amla	5g	1g	5g	1g
Beetroot	5g	—	5g	—
Curryleaves	3g	1g	3g	1g
Kalonji	3g	1g	3g	1g

Formulation of Hair Dye Paste for Application

For assessment, a specified quantity of each formulation was combined with a sufficient volume of distilled water to create a smooth, homogeneous paste. The paste was permitted to rest for a designated duration to guarantee the adequate release of dye molecules prior to application on hair samples.

Storage Specifications

All manufactured compositions were stored in hermetically sealed containers in a cold, dry environment, safeguarded from direct sunlight and moisture, to ensure stability and avert degradation.

Evaluation of Herbal Hair Dye

The aforementioned formulations were created and assessed for several characteristics, including organoleptic assessment, rheological evaluation, phytochemical evaluation, stability testing, and hair strand tests.

Organoleptic evaluation

Organoleptic evaluation is a qualitative assessment employed to analyze the sensory attributes of a hair dye product. This study assessed the hair dye samples for color, odor, texture, and appearance. The formulation's hue was visually assessed for consistency and intensity. Odour was evaluated to ascertain acceptability and detect any unpleasant scent. The texture and appearance were assessed through tactile and visual examination to confirm smoothness and homogeneity [7].

Rheological evaluation

Rheological qualities characterize the flow and deformation of a material under applied stress, and they are essential for comprehending the behavior of formulations such as herbal hair dye pastes, lotions, gels, and shampoos. Rheological assessment encompasses many tests such as bulk density, tapped density, angle of repose, Carr's index, and Hausner's ratio.

Bulk density is defined as the mass of a powder or granular substance divided by its whole volume, encompassing the voids between particles. A sufficient quantity of the sample (15 g) was run through a sieve, if required, to disaggregate agglomerates. The precisely weighed sample was carefully placed into a 100 mL measuring cylinder without compressing the powder. The powder bed was meticulously leveled without exerting pressure, and the uncompressed apparent volume was recorded to the nearest graded unit [8]. The sample's bulk density was subsequently computed using the equation provided below.

$$Bulk\ density = mass\ of\ powder / bulk\ volume\ of\ powder$$

Tapped density is the mass of a powder divided by the volume it occupies following mechanical tapping, which induces particle settlement and closer packing; to ascertain the tapped density, the identical measuring cylinder with the 15 g sample was subjected to mechanical tapping with a fixed drop of 14 ± 2 mm at a nominal frequency of 300 taps per minute until a stable volume was achieved. The final tapped volume was documented, and the tapped density was computed utilizing the appropriate formula [9]. Utilizing the acquired values of bulk density and tapped density, Carr's Compressibility Index and Hausner's Ratio were calculated to assess the flow characteristics of the powder blend.

$$Tapped\ density\ (g/ml) = Mass\ of\ powder\ (g) / Tapped\ volume\ (mL)$$

Using these density values, Carr's Compressibility Index and Hausner's Ratio were calculated using the standard formulas to assess the compressibility and flow characteristics of the formulation [10].

Carr's Compressibility Index (%) = (Tapped Density – Bulk Density)/tapped density * 100

Hausner's Ratio=Bulk Density/Tapped Density

The angle of repose is the maximum angle formed between the free surface of a powder pile and the horizontal plane when the powder is freely deposited onto a flat surface. It is the maximum angle at which a pile of powder can remain stable without collapsing. This is a straightforward empirical test utilized to evaluate the flowability of a powder [11].

The angle of repose was assessed using the fixed funnel method to analyze the flow characteristics of the powder blend. The powder sample was permitted to cascade through a funnel positioned at an appropriate height onto a flat surface until a conical pile was created. The height (h) of the pile and the radius (r) of the base were measured, and the angle of repose (θ) was determined using the formula

$\tan\theta = r/h$

Phytochemical evaluation

Molisch's Test for Carbohydrates: 2ml of extract were treated with 2–3 drops of Molisch's reagent, followed by the meticulous addition of strong sulfuric acid around the interior walls of the test tube. The emergence of a violet ring at the interface signified the existence of carbohydrates.

Fehling's Test (for Reducing Sugars): Equal quantities of Fehling's solution A and B were combined and introduced to the extract, thereafter heated in a water bath for several minutes. The emergence of a brick-red precipitate verified the existence of reducing sugars.

Ninhydrin Test (Amino Acids/Proteins): The extract was subjected to a few drops of ninhydrin reagent and subsequently heated. The manifestation of a purple or blue hue signifies the existence of amino acids or proteins. **Wagner's Test (Alkaloids):** Wagner's reagent was applied to the extract in a dropwise manner. The emergence of a reddish-brown precipitate signified the existence of alkaloids.

Ferric Chloride Assay (Phenolics/Tannins): A small quantity of ferric chloride solution was included into the extract. The emergence of a dark blue, green, or black hue signifies the presence of phenolic chemicals or tannins. **Alkaline Reagent Assay (Flavonoids):** The extract was subjected to a few drops of sodium hydroxide solution. The development of a vivid yellow hue that turned colorless following the introduction of dilute acid validated the existence of flavonoids.

Biuret Assay (Proteins): A sodium hydroxide solution and several drops of copper sulfate solution were added to the extract. The emergence of a violet or purple hue signifies the existence of proteins.

Froth Test (Saponins): The extract was diluted with distilled water and agitated vigorously for several minutes. The formation of steady, persistent froth suggested the presence of saponins.

Salkowski's Test (Steroids/Terpenoids): The extract was combined with chloroform, subsequently introducing concentrated sulfuric acid along the periphery of the test tube. The emergence of a reddish-brown ring signified the presence of steroids or terpenoids.

Keller–Killiani Test (Cardiac Glycosides): Ferric chloride was added to the extract of glacial acetic acid, followed by the meticulous addition of strong sulfuric acid along the test tube's walls [12]. The emergence of a brown ring at the interface signified the presence of cardiac glycosides [13].

Stability Tests

Test for shelf life

Shelf-life evaluation of herbal hair color formulations verifies product stability, microbiological safety, and preservation of efficacy under both accelerated and real-time circumstances, often ranging from 6 to 24 months in accordance with Drugs & Cosmetics Act regulations [14].

Test for pH

The pH of herbal hair dye formulation is essential for assessing its safety, compatibility with the scalp, and efficacy in coloring hair.

Test for Irritability

A patch test is a fundamental safety practice conducted on a limited skin area prior to the application of any topical preparation to assess potential allergic responses or irritation. This test is crucial for herbal hair color formulations containing constituents like henna, indigo, amla, shikakai, and methi to guarantee their safety for application on the scalp and skin [15].

Test for moisture content

The moisture content of the herbal hair dye mixture was assessed using the loss on drying method. Approximately 2–5 g of the sample was precisely measured in a pre-dried and pre-weighed petri plate. The sample was dehydrated in a hot air oven at 105°C until a stable weight was achieved. Subsequent to drying, the sample was chilled in a desiccator and subsequently reweighed [16]. The moisture content percentage was determined by the weight loss of the sample utilizing the usual procedure.

Moisture Content (%) = Initial Weight−Final Weight/ Initial Weight ×100

Test on hair strands

The pigmentation efficacy of the herbal hair dye mixture was assessed utilizing blonde hair strands. The formulations F1, F2, F3, and F4 were produced as pastes by immersing them overnight in tea decoction. The hair strands were cleansed with a gentle shampoo, completely rinsed with distilled water, and air-dried at ambient temperature before to the experiment. A sufficient amount of the prepared herbal hair color paste was evenly applied

to the hair strands with a brush and permitted to sit for a designated duration (60 minutes) at ambient temperature. Upon conclusion of the application period, the hair strands were meticulously rinsed with distilled water to eliminate surplus dye and subsequently air-dried. The dyed hair strands were subsequently assessed for color intensity, homogeneity, shade progression, smoothness, and color retention [17,18].

RESULTS AND DISCUSSION

Organoleptic evaluation

Organoleptic examination, also known as sensory evaluation, is conducted for all formulations (F1, F2, F3, F4). The factors assessed for organoleptic evaluation, including color, odor, texture, and appearance, together with the recorded results, are presented in Table 3.

Table 3: Organ oleptic evaluation of the formulations

Parameter	F1	F2	F3	F4
Colour	Dark Brownish	Greenish	Light Brownish	Greenish
Odour	Characteristic	Characteristic	Characteristic	Characteristic
Texture	Fine	Fine	Fine	Fine
Appearance	Powder	Powder	Powder	Powder

Based on the observations the formulations prepared were found to possess good colour, odour, texture and appearance

Rheological Evaluation

The rheological assessment of all four formulations for bulk density, tapped density, angle of repose, Carr's index and Hausner's ratio were conducted and represented in table 4. The herbal powder exhibited favorable flowability, with satisfactory measurements for bulk density, tapped density, and Carr's index, signifying ease of mixing and application. In general, a Carr's Index below 15% and a Hausner's Ratio below 1.25 indicate good flow properties suitable for formulation processing.

Table 4: Rheological evaluation of the formulations (F1, F2, F3 and F4)

Parameter	F1	F2	F3	F4
BulkDensity	0.75gm/cm^3	0.71gm/cm^3	0.78gm/cm^3	0.68gm/cm^3
Tapped Density	0.93gm/cm^3	0.88gm/cm^3	0.93gm/cm^3	0.83gm/cm^3
AngleofRepose	37.5	34.99	34.9	36.5
%Carr'sindex	14.3%	16.3%	15.1%	18%
Hausner'sratio	0.8	1.2	1.1	1.2

An appropriate Carr's index guarantees that the polyherbal powder will not aggregate into hard clumps during storage and facilitates uniform hydration during paste formulation. Hausner's ratio quantifies interparticle friction; an optimal Hausner's ratio facilitates homogeneous blending, efficient transfer into containers, and easy handling devoid of clogging or adhesion.

Phytochemical evaluation

The phytochemical examination of formulations F1, F2, F3, and F4 was conducted by making aqueous extracts of the powders. The findings demonstrate that the formulation encompasses a broad spectrum of phytochemicals obtained from the included herbal components. Each set of chemicals provides distinct functional advantages to the formulation. The documented outcomes are presented in Table 5.

Table 5: Phytochemical evaluation of the formulations

Parameter	F1	F2	F3	F4
Molisch'stest	+	+	+	+
Fehling'stest	+	+	+	+
Ninhydrintest	+	+	+	+
Wagner'stest	+	+	+	+
Ferricchloridetest	+	+	+	+
Alkalinereagenttest	+	+	+	+
Biuret test	+	+	+	+
Frothtest	+	+	+	+
Salkwoski'stest	+	+	+	+

Kellerkillanitest	+	+	+	+

Stability tests

Test for shelf life

All dry powder formulations (F1, F2, F3, and F4) were preserved in an airtight, sealed glass container at ambient temperature (25–30°C, standard humidity). No indications of contamination, such as alterations in color, odor, or texture, were detected in any of the formulations (F1, F2, F3, and F4). This implies that all formulations were stable and possess a decent shelf life. The results indicated no substantial deterioration and no microbial proliferation. This confirms the effectiveness of preservatives.

Test for pH

The pH levels of formulations F1, F2, F3, and F4 were assessed, and the findings are presented in Table 6.

Table 6:pH of herbal hair dye formulations F1,F2,F3 and F4

S. no	Formulation	pH
1.	F1	6.27
2.	F2	6.46
3.	F3	6.04
4.	F4	6.92

Test for Irritability

Irritability test for the formulations were conducted and were found to be safe for application.

Test for moisture content

Herbal hair dye powder formulations must have a loss on drying (LOD) of ≤2% w/w to ensure microbiological stability, prevent clumping, and sustain a 24-month shelf life. The computed outcomes for all four formulations (F1, F2, F3, and F4) are presented in Table 7.

The moisture content values within the specified range across all formulations demonstrate good optimization of the drying process and effective moisture regulation throughout the formulation of herbal hair dye powder.

Test on hair strands

The color intensity exhibited by all four formulations (F1, F2, F3, and F4) on a blonde hair strand (Figure 2) demonstrates that the formulations incorporating beetroot powder impart an exquisite aesthetic. The formulation F1, comprising commercial henna powder and beetroot powder, has demonstrated greater color intensity compared to the other formulations. Formulation F3, which includes natural henna and beetroot powder, exhibited commendable color intensity, however not to the extent of F1.

Table 7: LOD of herbal hair dye formulations (F1, F2, F3andF4)

S.No	Formulation	Losson Drying
1.	F1	0.84%
2.	F2	1.28%
3.	F3	0.89%

4.	F4	1.07%

Figure 2: Effect of herbal hair dye formulation on the blonde hair strand

DISCUSSION

This study illustrates the effective development and assessment of four polyherbal hair dye preparations (F1, F2, F3, and F4) utilizing both commercial and natural henna, with or without the use of beetroot powder. The systematic variation in composition facilitated the comparative evaluation of physicochemical attributes, stability, safety, and coloring performance [19,20]. The organoleptic assessment verified that all formulations exhibited satisfactory color, distinctive odor, smooth texture, and consistent powder appearance, signifying high patient acceptability and formulation quality. Rheological analyses demonstrated acceptable bulk density, tapped density, Carr's index, and Hausner's ratio, indicating favorable flow characteristics, manageable handling, and effective mixing during application.

These specifications guarantee that the powders maintain their free-flowing characteristics without clumping during storage. Phytochemical analysis revealed the existence of many bioactive compounds, including carbohydrates, proteins, alkaloids, flavonoids, tannins, and glycosides in all formulations, thereby affirming the medicinal potential and functional efficacy of the herbal components. Stability experiments revealed that all formulations maintained physical stability, exhibiting no alterations in color, odor, or texture, and showed no microbiological growth at room temperature, signifying excellent shelf life and preservation efficacy. The pH values (6.04–6.92) fell within a mild and scalp-friendly range, guaranteeing safety and compatibility. Irritability testing further validated that the formulations were non-irritant and well tolerated, affirming their appropriateness for topical administration. The moisture content (≤2% LOD) in all formulations demonstrated exceptional drying efficiency and microbiological stability. Hair strand analysis indicated that formulations with beetroot powder, especially F1, yielded increased color intensity, implying a synergistic interaction among the substances. Ultimately, F1 proved to be the most efficacious formulation for coloring performance and stability.

CONCLUSION

All four natural hair dye formulas were created and assessed. The amalgamation of henna and indigo constitutes the principal coloring system, while beetroot powder acts as a color intensifier. Concurrently, supportive herbs such as amla, hibiscus, shikakai, methi, kalonji, curry leaves, and bhringraj are included to augment pigment fixation, enhance hair texture, and foster scalp health. Formulation F1 was optimized from numerous formulations primarily due to its superior coloring efficacy on hair strands and other evaluation criteria. The formulated dry hair dye, with commercial henna powder and beetroot powder, effectively provides an aesthetically pleasing, natural hair color. This amalgamation of commercial henna and beets yields a consistent, uniform hue with substantial adherence and superior conditioning properties. In conclusion, the formulation demonstrates that combining standardized marketed henna with beetroot presents a straightforward, effective, and sophisticated solution for herbal hair coloring, yielding a cosmetically appealing, vibrant, and durable natural hue that endorses its potential as a commercially viable herbal hair dye product.

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