



“PREPARATION AND ASSESSMENT OF A SAFE AND EFFECTIVE SPRAY-ON TEMPORARY HERBAL HAIR COLORING”

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ABSTRACT:

Hair ageing is a common occurrence. There are many different kinds of hair colouring products available on the market composed of various chemical types that are either somewhat or significantly damaging to humans. This study proposed a temporary herbal hair dye spray that is easier to use than the conventional way of applying herbal hair color, offering a safe alternative to synthetic dyes that also offers additional advantages for long-term use for hair. The powdered forms of *Lawsonia inermis*, *Indigofera tinctoria*, *Embllica officinalis*, *Camellia sinesis*, *Bacopa monnieri*, *Acacia catechu*, and *Aloe vera* were extracted and combined with rosemary oil and lavender oil. Extracts were tested for conformation of their chemical constituents. To make it darker and help the hairs retain their colour, propylene glycol in a low concentration and iron fillings were also added to it. The dye was made in a thick liquid form that was simpler to spray and was later tested for skin sensitivity and colouring effectiveness. The mixture gives hair a dark brownish to black colour and without having any negative effects on the skin or hair because all of the ingredients are natural. The mixture is also simpler to use than the traditional method of mixing and applying powdered henna to hair because it is made to be used directly on hair.

Keywords: Herbal, hair dye, spray, henna, indigo, natural, hair, extracts, safe

1. INTRODUCTION

A typical suggestion of aging is slate hair. ⁽¹⁾ Greying is generally a progressive and unrecoverable process that's more frequently brought on by deteriorating melanocyte function than a reduction in the total number of melanocytes. As melanin-free hair scatters light, it appears white. ⁽²⁾

Hair dyeing is the process of changing the color of one's hair. The main reasons for this are cosmetics, similar to covering up hair that's considered more fashionable or desirable, or restoring the original hair color after it has been discolored by the hairdressing process. Men and women both color their hair. According to estimates, 40% of women regularly use hair colorings. ⁽³⁾ Dyes are colorings that are answerable in water and/ or an organic detergent, have a flyspeck size of lower than 0.01 m, and have an affinity for color substrates. ⁽⁴⁾

Synthetic color contains dangerous chemicals similar as ammonia, peroxide, p- phenylene diamine, diamino benzene, toluene 2,5-diamine, etc. These chemicals irritate the eyes and lungs, beget hair breakage and

dandruff, beget chemical becks, and can indeed beget cancer. ⁽⁵⁾ Temporary hair colorings absorb only in the cuticle and removes after 1 – 2 wetlands. Semi-permanent hair colourings only mask the melanin with colorings and washes out after 2 – 8 washes. They include 2% hydrogen peroxide and low attention to alkalizing chemical (frequently mono ethanolamine rather than ammonia). While endless hair colorings use ammonia as the alkalizing agent and contain up to 6% peroxide. This causes pH values to range from 9 to 10.5, which facilitates full penetration through the hair cortex. ⁽⁶⁾

Chemical family of aryl amines, which includes multitudinous potent mortal carcinogens. Toxicologists and epidemiologists have been concerned about the possible carcinogenicity of hair color chemicals for decades. ⁽⁷⁾

Paraphenylene diamine, a common component in numerous hair colorings, is well known to produce antipathetic contact dermatitis and Erythema multiforme (EM). ⁽⁸⁾ Bleaches that contain persulfate mariners (e.g., ammonium persulfate or potassium persulfate) causes breakage of the disulfide bond and leads to increased fineness due to porosity and lump. ⁽⁹⁾

For centuries, medicinal plants, also known as medicinal condiment, have been discovered and used in traditional drug practices, and are now being incorporated into color phrasings. These medicinal herbs can be used in the form of extracts, pulverized extracts, crude extracts, or derivations. Natural colorings are biodegradable, non-toxic, and non-allergenic, which makes them better for the terrain and use around humans because they do not contain carcinogenic factors set up in numerous synthetic colorings.





Herbs, as natural products, are allowed to be innately safe, effective, and fluently accessible, with no side effects. ⁽⁵⁾ Herbal hair colorings are available only in the form of dry powder but this study focuses on preparing more accessible and new way of herbal hair color i.e., herbal hair spray.






2. MATERIALS AND METHOD

2.1. MATERIALS USED AND THEIR SOURCE:

All the ingredients namely Henna (*Lawsonia inermis*), indigo (*Indigofera tinctoria*), amla (*phyllanthus emblica*), tea (*camellia sinesis*), brahmi (*bacopa monnieri*) and catechu (*acacia catechu*) were collected in powdered form from authorized store of local market. Rosemary oil and lavender oil were purchased from online authorized source.

Table 2.1 summary of ingredients used in herbal hair dye formulation ⁽¹⁰⁾

S. No.	Common and Botanical name	Picture	Chemical Constituents	Uses
1	Henna: <i>Lawsonia inermis</i>		Lawsone, flavonoids, gallic acid, tannins	Coloring agent, antifungal
2	Indigo: <i>Indigofera tinctoria</i>		Indigotin, indirubin, flavonoids, tannins	Coloring agent, promote hair growth, prevent infection
3	Amla: <i>Emblica officinalis</i>		Vitamin C, tannins, antioxidants	Maintain hair color, promote hair growth, strengthen hairs
4	Tea: <i>Camellia sinesis</i>		Isoflavone, flavonoids, tannins, amino acids	Darken the hair, mordant, conditioner, antioxidants

5	Brahmi: Bacopa monnieri		Bacoside A and B, flavonoids, saponins	Conditioner, promote hair growth
6	Catechu: Acacia catechu		Catechin, tannic acid, catechu red	Astringent, antifungal
7	Rosemary oil: Rosmarinus officinalis L.		Cineol, camphor, borneol, pinene	Improve hair growth, prevent baldness, makes hair shinier, preservative
8	Lavender oil: Lavandula Angustifolia		Linalool, linalyl acetate, cineole, camphor	Promote hair growth, antimicrobial, provide shine to hairs.
9	Aloe Vera: Aloe barbadensis miller		Aloin, aloe emodin, glycosides	Mordant, nourishes hair

2.2. INSTRUMENTS:

1. Soxhlet Apparatus
2. Heating Mantle
3. Electric water bath
4. TLC plate
5. Hot air oven
6. UV Chamber
7. Digital pH meter
8. Brookfield viscometer

2.3. PREPARATION OF HERBAL EXTRACTS:

2.3.1. Henna extract: 100 gm of henna powder was mixed with 1000 ml of 70% ethanol and kept for maceration for 12 hours. After 12 hours extract was filtered with the help of Buchner funnel and whattman filter paper and dried at 50°C. ⁽¹¹⁾ 40 gm of thick henna extract was obtained.

2.3.2. Indigo extract: Indigo powder was extracted with ethanol under reflux. 50 gm of indigo powder was added to 300 ml ethanol. Mixture was filtered by Buchner funnel and whattman filter paper, Solvent was dried and concentrated using evaporator at 50°C. ⁽¹²⁾ 15 gm of thick indigo extract was obtained.

2.3.3. Amla extract: 50 gm of amla powder was extracted using 200ml of 50% ethanol in Soxhlet apparatus. Extract was concentrated under reduced pressure to yield syrup mass. Extract was stored in air tight container in a cool place. ⁽¹³⁾ 20 gm of thick amla extract was obtained.

2.3.4. Tea extract: 50 gm of tea was mixed with 500 ml of hot distilled water in a closed vessel for 1 day. Mixture was filtered by Buchner funnel and whattman filter paper, Solvent was dried and concentrated. ⁽¹⁴⁾ 20 gm of thick tea extract was obtained.

2.3.5. Brahmi extract: 50 gm of brahmi powder was extracted using 200 ml of ethanol in Soxhlet apparatus. Extract was concentrated and dried. ⁽¹⁵⁾ 10 gm of thick brahmi extract was obtained.

2.3.6. Catechu extract: 20 gm of catechu powder was added in 180 ml of distilled water with an initial temperature of 80°C. The solution was taken in a conical flask and wrapped with aluminium foil. Shake the flask at incubator shaker for 10 minutes at 60°C. Rest it for 1 day and filter with Whatman filter paper. Evaporate the filtrate and concentrated extract was obtained. ⁽¹⁶⁾ 10 gm of thick catechu extract was obtained.

2.3.7. Aloe vera Extract: Fresh Aloe vera leaves were collected and washed. Outer green part of leaf was peeled off and inner

white mass were grinded and filtered. Filtrate was subjected to evaporation to 1/10th of its volume. ⁽¹⁷⁾

2.4. PRE-FORMULATION STUDIES:

2.4.1. Preliminary Phytochemical Screening of plant extracts

The analysis of various phyto-chemicals, such as carbohydrates, saponins, oils, lipids, flavonoids, terpenoids, alkaloids, etc., was done using phytochemical screening. Plant samples extracted with methanol. ⁽¹⁸⁾ Results are shown in Table 3.1

2.4.1.1. Test for Carbohydrate (Reducing Sugars) (Fehling's Test)

The extract was treated with 5.0 ml of Fehling's Solution and kept in boiling water bath. The formation of red or yellow precipitate indicates the presence of carbohydrate.

2.4.1.2. Test for Saponins (Honeycomb Test)

0.5 mg extract was taken in a test tube and 5 drops of 5% NaHCO₃ was added and shake for 3 minutes. Formation of honeycomb like froth indicates presence of saponins.

2.4.1.3. Test for Terpenoids (Salkowski Test)

5 ml of extract was mixed in 2 ml of chloroform and concentrated H₂SO₄ was added to form a layer. A reddish-brown color of interface indicates the presence of terpenoids.

2.4.1.4. Test for Alkaloids (Dragendroff's Test)

The plant extract was mixed with a few drops of acetic acid and then Dragendroff's reagent was mixed well. Formation of orange red precipitate indicates the presence of alkaloid.

2.4.1.5. Test for Tannins and Phenolic (FeCl₃ Test)

1 ml of extract was mixed with 2 ml of 5% FeCl₃ solution. Formation of Blue color indicates the presence of Tannins and phenolic compounds.

2.4.1.6. Test for Amino Acids and Proteins

1 ml of extract was heated with 2 drops of freshly prepared ninhydrin reagent. Formation of blue color indicates the presence of Amino acid and proteins.

2.5. ANALYTICAL TEST (THIN LAYER CHROMATOGRAPHY):

Preparation of Slurry: Required amount of Silica gel G was mixed to sufficient amount of distilled water to make a thick slurry.

Preparation of TLC plates: TLC plates (20 cm x 20 cm) was washed with soap and water and then dried in oven. Slurry is then poured on the plates. Slurry is sprayed evenly on the surface of glass plates. Thickness of the adsorbent layer was around 0.1 - 0.25 mm.

Activation of TLC plates: Prepared TLC plates were placed in oven and heated for 30 minutes at 110°C to remove adsorbed water molecules. After 30 minutes the plates were removed and after cooling, they were marked 1 cm from bottom. Sample was applied on the TLC plates with the help of capillary tube. Spots were air dried and then the plate is suspended in the chromatography jar containing solvent and was allowed to run to 3/4th position. TLC plates then examined under UV chamber at 365 nm.

Different solvents system used are:

For Henna: Toluene : Ethyl acetate : Formic Acid

5.5 : 4 : 0.5

For Amla: Toluene : Methanol

8 : 2

For Indigo: Toluene : Methanol

8 : 2

For Tea: Butanol : Glacial acetic acid : Water

4 : 1 : 5

The presence of carbohydrate, flavonoids, tannins, alkaloids and amino acids have been confirmed by thin layer chromatography showing spots with Rf values of 0.78, 0.75, 0.56, 0.63 respectively.

2.6. FORMULATION OF HAIR DYE SPRAY:

Table 2.2 Formulation of Hair Dye

Sr. No.	Ingredients	Formulation (F1)	Formulation (F2)	Formulation (F3)	Formulation (F4)
1	Henna	2 ml	2 ml	3 ml	4 ml
2	Indigo	0.5 ml	1 ml	3 ml	8 ml
3	Amla	0.5 ml	1 ml	3 ml	3 ml
4	Tea	0.5 ml	1 ml	3 ml	2 ml
5	Brahmi	0.5 ml	0.5 ml	1 ml	2 ml
6	Catechu	0.5 ml	0.5 ml	1 ml	2 ml
7	Rosemary oil	5 drops	5 drops	5 drops	5 drops
8	Lavender oil	5 drops	5 drops	5 drops	5 drops
9	Aloe Vera	1 ml	1 ml	1 ml	2 ml
10	Propylene glycol (1%)	-	-	-	2 ml
11	Iron filings	-	-	-	2 gm

2.7. EVALUATION OF HAIR DYE SPRAY:

2.7.1. Organoleptic Evaluation

- Color
- Odour
- Apperance

Organoleptic characteristics like color and odour of the prepared formulation was tested along with the other special features like appearance.

2.7.2. Physio-chemical evaluation

- pH
- Viscosity

The physical and chemical properties of the dye were determined by examining pH and viscosity of the formulation. pH was measured using Digital pH meter. Viscosity was measured using BROOK FIELD'S VISCOMETER at 20 rpm using spindle 64.

2.7.3. Patch Test

It is necessary to check the sensitivity of the formulation on the skin. This was done by applying the formulation behind the ear or internal ear in area of 1 cm² and let it dry. Any signs of itching, irritation or erythema within 3 hours of application were noted.

2.7.4. Study on dyeing effect

All the formulations were sprayed on the hair samples and let it dry for 1 hour. After complete drying of the hairs, hairs were washed with only water and dried them again. After drying of hairs, the color fastness and dyeing effect on hairs were observed.

2.7.5. Stability Test

Stability testing was performed by storing the selected formulation for a period of 1 month at different temperature mainly at room temperature and at 35°C to check any changes in color, odour, appearance, pH and viscosity of the formulation.

3. RESULT AND DISCUSSION

3.1. Phyto-chemical Evaluation Result

Table 3.1 Phyto-chemical Evaluation of Herbal Extracts

Sr No.	Phytoconstituents	Henna	Indigo	Amla	Tea	Brahmi	Catechu	Aloe
1	Carbohydrates	+	+	+	+	+	-	+
2	Saponins	-	-	-	-	+	-	+
3	Terpenoids	+	+	-	-	+	-	-
4	Alkaloids	-	-	+	+	+	+	-
5	Tannins and Phenolics	+	+	+	+	+	+	+
6	Amino acids and Proteins	-	-	+	+	-	-	-

- To determine whether phyto-chemicals were present in all of the produced extracts, phyto-chemical screening was conducted. Every extract aside from catechu contains carbohydrates, according to Fehling's test, which was used to detect their presence.

- Only Brahmi and aloe had saponins, according to the results of the honeycomb test used to identify the saponins. Terpenoids were detected in the extracts using the Salkowski test, which reveals that the Brahmi, indigo, and henna extracts all contain terpenoids.
- According to Dragendorff's alkaloids test, alkaloids can be found in amla, tea, Brahmi, and catechu. All of the extract tested positive for tannins and phenolic compounds using ferric chloride. Only amla and tea exhibit the existence of proteins and amino acids.

3.2. Physio-chemical Evaluation Result

Table 3.2 Physio-chemical Evaluation

Sr No.	Parameters	F1	F2	F3	F4
1	Color	Light Brown	Dark Brown	Brownish Black	Blackish Brown
2	Odour	Characteristic	Characteristic	Characteristic	Characteristic
3	Appearance	Liquid	Liquid	Liquid	Liquid

- The physical characteristics of prepared formulations are mentioned in the Table 2.2 which shows that the colour of formulation further improved to blackish brown after addition of iron fillings.

3.3. Dyeing Effect on Human Hair



Figure 3.1 Dyeing effect of herbal hair dye

Table 3.3 Study on dyeing effect on human hair with herbal hair dye

Dyeing Effect	After 60 min			
	F1	F2	F3	F4
Appearance of hair	No damage	No damage	No damage	No damage
Colour of the hair	Light brown	Dark Brown	Blackish Brown	Black

- All the formulations F1, F2 and F3 shows light color on human hair that washes off after hair wash. In formulation F4 dye shows darker shade of color after addition of iron fillings as given in ⁽¹⁹⁾ also adding 1% propylene glycol enhances dyeing effect of formulation. ⁽²⁰⁾ Also, the color starts to

retain on hair after washing hairs and color does not fade away. Hence, formulation F4 is selected as the final formulation.

3.4. Patch Test Result

Table 3.4 Patch Test

Sr No.	Parameters	Result
1	Swelling	Negative
2	Redness	Negative
3	Irritation	Negative

○ Patch test shows negative results for all the measured parameters which indicates that the dye is safe to use.

3.5. Organoleptic Evaluation Result

Table 3.5 Organoleptic Evaluation of Herbal Hair Dye

Sr No.	Parameters	Result
1	Ph	7.5 ± 1
2	Viscosity (cP)	60

- Dye with pH between 5.5-8 tends to form a temporary formulation for hair. Formulations at lower pH was unable to sustain on hair. Therefore, after addition of iron fillings and propylene glycol the colour shade of the dye gets dark and it also starts to retain on hair.
- Hence, slightly alkaline pH is necessary for dye to stain hairs. The formulation after addition of iron fillings shows pH of 7.5 ±1 which was measured by using digital pH meter.
- To ensure retain ability of solution on hair, the dye was made little viscous and the viscosity was measured by Brookfield viscometer which shows value of 60 centipoise at 20 rpm using spindle 64.



Figure 3.2 Digital pH meter



Figure 3.3 Brookfield Viscometer

3.6. Stability Result

Stability test was performed for duration of 3 months at different temperature and the formulation was found to be stable during this time period. Since it contains antioxidant like amla and other natural ingredient and it is free from any artificial preservative, this leads to increased shelf life without any harmful chemicals in it.

4. CONCLUSION

The results of this study suggest that a natural hair dye in the form of a hair spray can be made to produce hair colours ranging from black to dark brown by adjusting the proportions of henna, indigo and amla. Since all of the substances are natural, there are no known side effects for the skin. Also, this composition is stable at room temperature and all of the ingredients are 100 percent biodegradable.

Propylene glycol in low concentrations and the inclusion of iron fillings boost the colour shade of henna, and the dye exhibits better absorption at pH levels higher than 6.5. In addition to colouring hair, the dye has a variety of other advantages for hair, including strengthening (Amla, Henna), conditioning (Brahmi, Tea), avoiding alopecia (Lavender oil and Rosemary oil), and minimising hair loss.

We found that effective property of herbal hair spray and further studies are needed to be performed to explore more benefits of this herbal hair spray and further changes that can be made in this formulation.

Consequently, it can be said that this formulation is wonderful for satisfying the needs of the worldwide market, safe to use, and easier to apply. This study can be helpful for upcoming researchers in developing a spray on herbal hair dye and its evaluation which can be claimed for their efficacy with scientific data.

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