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Formulation and evaluation, comparison of herbal hair dye with marketed formulation

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Abstract

Herbal products are being preferred due to the advantages in contrast to the synthetic one which has adverse effect on human health because of harsh chemicals like paraphenylene diamine. The word "Herbal" is a symbol of safety. Thus, our current research was aimed to prepare the herbal hair dye using the herbal extract from the diverse powders of herbals such as, *Lawsonia inermis*, *Aloe vera*, *Phyllanthus emblica*, *Thea sinensis*, *Trigonella foenum-graecum*, *Eclipta alba*, *Acacia catechu*, *Indigofera tinctoria*, and *Terminalia bellirica*. Three different formulations were made by mixing the varying proportion of extract from henna and indigo at the ratio of 1:0, 2:1 and 1:2, respectively. These different combinations of formulation were further evaluated for dyeing effect on unpigmented hair. The results with F3 formulation contain the extract of henna and indigo at a ratio of 1:2 that gave dark brown to black colour to hair, resembling natural hair colour. From our pharmaceutical evaluation studies, the formulation exhibited the better dyeing effect, free from skin irritation, free of obnoxious odour and great retention capacity, benefits of promotion of hair growth, suitable for all age group and ecofriendly on comparison with marketed synthetic dye formulation.

1. Introduction

Colors derived from plants and animals without any chemical processing are known as natural dyes. Herbal remedies that contain phytochemicals derived from a variety of botanical sources, which affect and supply nutrition for good skin and hair (Nupur, 2021; Sumit *et al.*, 2012). Hairs are an important part of human beauty. People are using herbs for cleaning, beautifying and hair growth since the ancient era (Dubey *et al.*, 2004). There are a variety of medicinal plants with possible hair effects that have been used for centuries around the world and are now incorporated into dye formulations. These medicinal herbs can be utilised as extracts, powdered extracts, crude extracts, or derivatives (Latha *et al.*, 2021; Vijayalakshmi *et al.*, 2018; Gangurde *et al.*, 2013).

Researchers have been searching for novel medications in nature since the dawn of mankind (Chanchal *et al.*, 2017). Plant products have been used in phytomedicine from the beginning of time. Around 80% of the world's population uses traditional medicines based on plant extracts for primary healthcare. Almost 90% of prescriptions in traditional systems of Unani, Ayurveda, Homeopathy, and Siddha were based on drugs derived from plants (Fathima *et al.*, 2011; Amsa *et al.*, 2014). Plant-based medicines are readily available, less priced, safe, and effective, with little side effects. Saffron and indigo were also utilised by the ancients to give the dye a desired function

(Kumar *et al.*, 2016). The colour that is currently available contains ingredients that are amino compounds (4- amino-2-hydroxytoluene and m-aminophenol). Colorants such as titanium dioxide and iron oxide are also commonly employed in the process (Mallya *et al.*, 2015). Prolonged use of such dye-containing substances on natural hair has numerous negative consequences, including skin irritation, erythema, hair loss or damage, and skin cancer (Nilani *et al.*, 2010). Synthetic hair dyes contain harmful and hair-damaging chemicals such as paraphenylene diamine, ammonia, and paraben. Many people are switching to natural hair colouring methods as they become more aware of their side effects (Prabhu *et al.*, 2012). Natural hair dyes are a safer alternative because they are made up of plant-based ingredients rather as harsh chemicals (Pal *et al.*, 2018).

Natural hair dye gives our hair a natural black colour without harming it. Even after repeated colouring, our hair remains healthy as a result of this. Because traditional hair colouring procedures based on natural or synthetic colourants have limitations, this study aimed to develop a hair dye based on crude medicines with good colouring properties that is both safe and ready to use (Mahale *et al.*, 2000; Kadambari *et al.*, 2009).

2. Materials and Methods

2.1 Plant source

The powder of henna (*Lawsonia inermis*), indigo (*Indigofera tinctoria*), aloe (*Aloe vera*), amla (*Phyllanthus emblica*), tea (*Thea sinensis*), fenugreek (*Trigonella foenum-graecum*), bhringraj (*Eclipta alba*), black catechu (*Acacia catechu*), myrobalan (*Terminalia chebula*), were procured from Ayurvedic shop in Erode, Tamilnadu, India.

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Description of the ingredients of the herbal hair dye (Gediya *et al.*, 2001; Chaudhary *et al.*, 2010; Pandey *et al.*, 2010; Kaur *et al.*, 2002)

S.No.	Common and botanical name	Picture	Chemical constituents	Uses
1.	Henna: <i>Lawsonia inermis</i>		Lawsone, flavonoids, gallic acid	Coloring agent, antifungal
2.	Indigo: <i>Indigofera tinctoria</i>		Glycosideindan, indigotin, indirubin flavonoids, tannins	Colouring agent, promote hair growth
3.	Hibiscus: <i>Hibiscus rosa-sinensis</i>		Alkaloids, tannins, anthocyanin, beta carotene	Demulcent, lower blood pressure
4.	Amla: <i>Phyllanthus embilica</i>		Rich in vitamin C tannins,	Antioxidant, maintains hair colour
5.	Aloe: <i>Aloe vera</i>		Anthroquinone glycoside, tannins	Natural mordant nourishes hair
6.	Bringraj: <i>Eclipta alba</i>		Flavonoids, triterpines, steroids	Natural hair tonic
7.	Black catechu: <i>Acacia catechu</i>		Catechins, catechu tannic acid, catechu red	Astringent, antifungal
8.	Myrobalan: <i>Terminalia chebula</i>		Tannins, ellagicacid, chebolic acid	Mordant, hair softening
9.	Tea: <i>Camellia sinensis</i>		Isoflavone, aminoacid, Tannins, carotenoids.	Conditioner, mordant, darken the hair, and antioxidant

2.2 Preparation of herbal extracts

100 gm each of all the herbal powder was taken separately and boiled with 100 ml of distilled water for 1 h and the extract was filtered and evaporated to dryness.

2.3 Phytochemical evaluation

The prepared herbal extract was subjected to phytochemical screening to reveal the various phytoconstituents as carbohydrates, lipids, alkaloids and sugars, *etc.* (Bhawana *et al.*, 2021; Kokate *et*

al., 2008; Wallis *et al.*, 2002., Malik *et al.*, 2020). The extract was dissolved individually in 5 ml of water and filtered; the filtrates were used to carry out the chemical test and the result was shown in Table 1.

2.4 Formulation of herbal hair dye

All the dried herbal extracts were taken as per the quantities mentioned in Table 2 and it was continuously mixed with water to prepare uniform pastes.

2.5 Evaluation of the herbal hair dye

The formulated herbal hair dye was evaluated for various parameters, such as organoleptic, physicochemical, rheological and phytochemical evaluation.

2.5.1 Organoleptic evaluation

Organoleptic characteristics for various sensory characters like color, taste, odour, and special features, like touch, texture and appearance, etc., was carefully identified (Evans *et al.*, 1997; Padmini *et al.*, 2013) and was illustrated in Table 3.

2.5.2 Physicochemical evaluation

The physical and chemical features of the herbal hair dye were evaluated to determine the pH, moisture content, and ash value (Kokate *et al.*, 1994; Meenaprabha *et al.*, 2021; Sagar *et al.*, 2005; Singh *et al.*, 2020). All these findings are mention in Table 4.

- i. **pH:** The pH of formulated herbal hair dye was determined using pH meter.
- ii. **Moisture content:** A method commonly used for moisture content determination is the loss on drying method or LOD. The crude drugs heated at 105°C to constant weight and calculate the total loss of weight.
- iii. **Total ash value:** The 2 gm of sample was taken in a silica crucible then ignited by gradually increasing the heat to 400°C until it appeared white indicating absence of carbon. It is then cooled in a desiccator and total ash of air dried material is calculated.

2.5.3 Rheological evaluation

The parameters like untapped or bulk density, tapped density, the angle of repose, Hausner's ratio, and carr's index were observed (Gennaro *et al.*, 2000; Sneha *et al.*, 2014; Kokare *et al.*, 2014; Puranik *et al.*, 2012; Taha *et al.*, 2019) and it was shown in Table 4.

- i. **Angle of Repose:** Angle of repose was determined using the funnel to estimate the flow behavior of the sample and it was determined by using the formula;

$$\tan \alpha = H/R$$

where, α = angle of repose; H = height of the pile of powder; R = radius of the pile of powder.

- ii. **Determination of bulk density and tapped density:** Bulk density and tapped density was determined by placing the cylinder containing sample (W1) in bulk density apparatus and the volume was noted as V1 (ml), then the apparatus adjusted for 100 tapping and operated. The volume occupied by the powder as V2 (ml) was recorded.

$$\text{Bulk Density} = \text{Mass/Bulk volume} = W1/ V1 \text{ g/ml}$$

$$\text{Tapped Density} = \text{Mass/Tapped volume} = W1/ V2 \text{ g/ml}$$

- iii. **Hausner's ratio:** Hausner's ratio is the ease of index of powder flow and calculated by using following formula:

$$\text{Hausner's ratio} = \text{Tap density/Bulk density}$$

- iv. **Carr's index:** Per cent compressibility of blend was determined by Carr's compressibility index, which was calculated by using following formula:

$$\text{Carr's index} = \frac{\text{Tap density} - \text{Bulk density}}{\text{Bulk density}} \times 100$$

2.6 Skin sensitivity test

Because herbal hair dyes should not cause skin sensitization or irritation when applied to hair, the samples were subjected to a skin irritation research. The skin irritation was calculated using the Draize modified scoring technique every 24 h for seven days after the colours were applied, and the skin irritation investigations were conducted on healthy rabbits (Regupathi *et al.*, 2016). The experimental protocol was approved by Institutional Animal Ethic Committee. (688 /2/CPCSEA). The rabbits' dorsal surface (50 cm²) was cleaned and the hair was shaved away. With rectified spirit, the skin was washed. Samples of herbal hair dye were applied to the skin. The skin was monitored for six days to check the skin irritation, redness, and edoema production. Table 4 shows the results of testing to look for indicators of irritation.

2.7 Stability test

Stability testing of the prepared formulation was performed by storing it at different temperature conditions for the time period of one month. The packed formulation were stored at different temperature conditions, viz., room temperature and 35°C and were evaluated for the physical parameters. The prepared dye formulations were evaluated for the physical parameters like colour, odour, pH, texture, and smoothness (Arora *et al.*, 2019).

2.8 Studies on dyeing effect

Each formulated dye (F1-F3) was applied to the gray hair which was collected from human volunteers. The colouring effect of formulated dye was observed After 30 and 60 min. The hairs were washed with herbal shampoo and dried at sun light. The fastness property and dyeing effect on gray hair was observed and the results were shown in Table 5.

2.9 Comparison of dyeing effect with marketed brand

The dyeing effect of formulated dye was compared with synthetic hair dye marketed as black henna was used as standard. The colour comparison was noted in different time duration such as 30 and 60 min and was reflected in Table 5.

3. Results

The Table 1 highlight of various phytoconstituents present in the herbal formulation. Henna and Indigo contain a pigment used as a colouring agent, tannin present in the tea, and aloe used as a mordant and it enhance colour intensity of hair, in addition to it promote hair growth and act as emollient. Amla and Hibiscus contain vitamins, aminoacids, and flavonoids, which together form a powerful antioxidant, antidandruff and it is beneficial for smooth and silky hair, whereas, Hibiscus used as good conditioner.

3.1 Evaluation of the herbal hair dye

The formulated herbal hair dye was evaluated for various parameters, such as, organoleptic, physicochemical, rheological, and phytochemical evaluation.

The preparation of herbal hair dye Henna and Indigo were chosen for natural colorant and it was brownish black, blackish brown in colour and appears as a smooth powder with pleasant odour.

Table 1: Phytochemical evaluation of herbal extract

S.No.	Phytoconstituents	Henna	Indigo	Hibiscus	Amla	Aloe	Bhringraj	Black catechu	Myrobalan	Tea
1	Alkaloids	-	-	+	+	-	+	+	-	+
2	Glycosides	+	-	+	-	+	-	-	+	-
3	Tannins	+	+	-	+	+	-	+	+	+
4	Flavonoids	+	+	+	+	-	-	+	-	+
5	Terpenoids	+	+	+	-	-	+	-	+	-
6	Carbohydrates	+	+	+	+	+	+	-	+	+
7	Aminoacids	-	-	+	+	-	+	-	-	+
8	Carotinoids	+	-	+	-	-	-	-	-	+
9	Saponins	-	-	+	-	+	+	-	+	-

Table 2: Formulation composition of herbal hair dye

S.No.	Ingredient	Formulation (F1)	Formulation (F2)	Formulation (F3)
1	Henna	35 g	20 g	10 g
2	Indigo	-	10 g	20 g
3	Hibiscus	5 g	5 g	5 g
4	Amla	2 g	2 g	2 g
5	Aloe	2 g	2 g	2 g
6	Bhringraj	2 g	2 g	2 g
7	Black catechu	-	5 g	5 g
8	Myrobalan	-	2 g	2 g
9	Tea	2 g	2 g	2 g

Table 3: Organoleptic evaluation of herbal dye

S.No.	Parameters	F1	F2	F3
1	Colour	Reddish brown	Brownish black	Blackish brown
2	Odour	Characteristic	Characteristic	Characteristic
3	Texture	Smooth	Smooth	Smooth
4	Appearance	Powder	Powder	Powder

Table 4: Physicochemical evaluation parameters

S.No.	Parameters	F1	F2	F3
1	Bulk density	0.32	0.31	0.30
2	Tapped density	0.51	0.50	0.45
3	Angle of repose	27.55	27.65	26.54
4	Corr's index	1.22	1.20	1.10
5	Hausner's ratio	1.24	1.36	1.30
6	PH	6.7	6.9	6.5
7	LOD	1.7	1.9	1.5
8	Ash value	0.18	0.16	0.17
9	Irritancy test	Nil	Nil	Nil

Table 5: Study on dyeing effect on human hair with herbal hair dye

Dyeing effect	After 30 min			After 60 min		
	F1	F2	F3	F1	F2	F3
Appearance of hair	No damage	No damage	No damage	No damage	No damage	No damage
Colour of the hair	Orange	Red	Reddish brown	Brown	Brownish black	black

Table 6: Study on dyeing effect on human hair with marketed brand

S.No.	Dyeing effect	After 30 min	After 60 min
1	Appearance of hair	Hair damage	Cortex damage
2	Colour of the hair	Black	Dark black

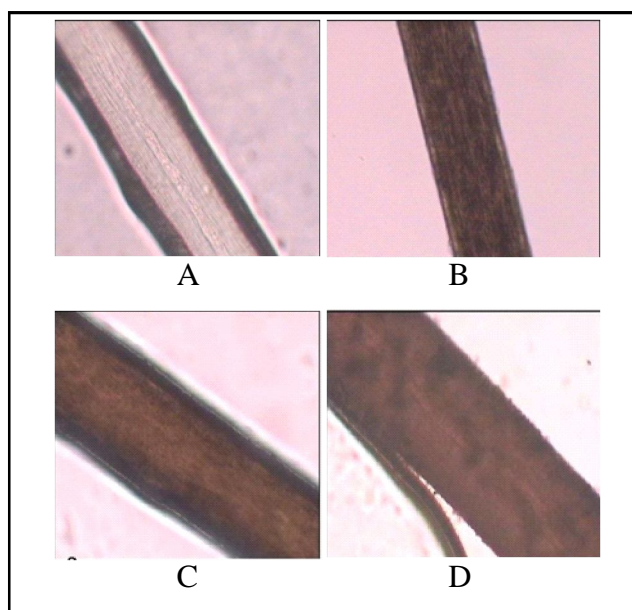
The Table 4 mentioned that the low moisture content was found that will enhance the flow property and prevents the microbial contamination during storage, the ash value was found to be 0.17 and it was nominal. Indicating the presence of inorganic radicals in significant amount (pH 6.5), which was found to neutral and it was suitable of different scalp types. However, the skin irritation study result showed that all the formulations (F1-F3) were free from allergic reactions to rabbit skin, no itching, redness, and rashes have been observed for all the formulations.

3.2 Stability test

Stability test was performed for approximately one month period at different temperature to observe the changes in its colour, odour, texture, appearance, pH. The formulations were found to be stable during this period. Thus, the result conclude that the prepared formulations were could be easily stored and used at any stability conditions. Since it is a natural herbal based formulation, *i.e.*, it is free from harsh chemical, preservatives and artificial colors. Moreover, the moisture content was low and there are minimal possibilities of the deterioration of the formulation, this leads to an increased shelf life due to goodness of natural ingredients.

From the results shown in above Table 5, the varying colour intensity was observed with different formulation. The formulation was prepared with varying proportion of Henna and Indigo along with above mentioned powder and helps to create various colour shades. Good dyeing effect was obtained with formulation (F3) had the ratio of Henna and Indigo (1:2), that gave black colour to the hair. The glimpse was shown in Figure 1.

The colour intensity was compared with that of marketed product which contains PPD in their formulation and the result shows good dyeing effect within 30 min, but it cause hair damage and skin irritation because of harsh chemicals (Figure 2).

**Figure 1: Dyeing effect of herbal hair dye on human gray hair.****Figure 2: Comparison of dyeing effect of herbal hair dye with marketed brand on human gray hair.**

- Microscopic features of gray hair treated with herbal hair dye showing no damaged cortex after 30 min.
- Microscopic features of gray hair treated with herbal hair dye showing no damaged cortex after 30 min.
- Microscopic features of gray hair treated with marketed brand showing no damaged cortex after 30 min.
- Microscopic features of grey hair treated with marketed brand showing damaged cortex after 60 min.

4. Discussion

The herbal hair color that has been developed has all of the benefits of natural components. Aside from acting as a hair colour, this composition also functions as a hair growth booster, hair nourisher, conditioner and antidandruff agent owing to the right combination of natural herbs. Henna, the basic powder, serves as a worldwide hair color, as it is utilised for its dyeing capabilities all over the world. It also helps to remove excess sebum from the scalp while also conditioning the hair. Bhringraj helps to improve blood circulation at the base of the hair (Rashmi *et al.*, 2018). Amla is high in vitamins A, C, D, and K that combine to make a potent

antioxidant. This antioxidant is likely the main factor your hair requires to remove oil accumulation from the hair, clear pores, kill disease bacteria, and stimulate hair growth. Hair fall, flaking, and grey hair may all be readily controlled by utilising hibiscus flower juice on a routine basis. Amla's high vitamin C content aims to mitigate early gray hair. It is an excellent hair booster and dandruff cure. Complementary formulations of carotenoids, flavonoids, and tannins are included in the herbal hair color composition, which assist to the colouring impact on human hair while also protecting it from photodamage. The leaves of *Camellia sinensis* and *Hibiscus rosa-sinensis* are a useful source of raw material for natural colouring because they are abundant in carotenoids, vitamins, flavonoids, and isoflavone. *Aloe vera* gel, which contains polysaccharide and moisture, serves as a natural mordant and also replenishes dry aged skin and hair, as well as preventing photodamage (Ashawat *et al.*, 2008; Nilani and Saravanan, 2010). The efficacy data indicates that by adjusting the proportions of Henna and Indigo, an appropriate black colour for hair can be achieved. The colour intensity increases with repeated application of this formulation, and the colour lasts for around 20 days even after regular washing and exposure to sunlight. This effect can be due to the tannins in the formulation, which function as mordants and help to increase the dye's colour intensity.

5. Conclusion

According to our study findings, an appropriate black colour for hair may be obtained by varying the proportions of Henna and Indigo. Hair colourant absorption was greatest at a pH of 6.5 - 6.9 and moreover, the use of the Henna and Indigo increased the colour intensity. This natural hair dye has the benefit of causing no skin damage, erythema development, or edoema. It is made entirely of water soluble plant elements, and as a result, it has no unpleasant odour. As well, both the raw components and the finished product are 100 % biodegradable and these compositions are stable at room temperature. This natural herbal hair colourant is excellent for people of all age group and the formulation and processing methods are ecosustainable. Hence, we discovered the beneficial qualities of the natural herbal dye in our study; however, an additional research is needed to discover more helpful advantages of this herbal hair pack. Thus, the herbal formulations are in popular to satisfy the expectations of an expanding global market.

Conflict of interest

The authors declare no conflicts of interest relevant to this article.

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