In the present investigation, attempts were made to make a powder herbal hair dye that gives dark brown to black colour to hair, resembling natural hair colour with better dyeing effect and greater retention capacity on comparison with marketed herbal hair dye formulation. Most of the marketed herbal hair dye formulations contain para-phenylenediamine at 20-25% concentrations which is the main ingredient of commercial synthetic dyes. It is known to trigger allergic skin reactions in many people. Ayurvedic powder hair dye devoid of any synthetic agent was prepared in present research work and evaluated for dyeing efficiency. Different combinations of powdered leaves, fruits of plants like Madayantika, Bhringraj, Amla and Nilini were evaluated as hair dyes. The polyherbal hair dye compositions containing large proportion of Nilini, Mehandi and 5% Bhringraj and Amla, blended with 5% loha bhasma were prepared and evaluated for physicochemical parameters like particle size, colour, pH, angle of repose and bulk density. Ayurvedic polyherbal powder hair dye of present investigation is semi-permanent in nature and exhibit better dyeing efficiency than marketed herbal hair dye.

Key words: Herbal hair formulation, Hair dyeing, Nilini, Bhringraj, Madayantika, Loha bhasma.

INTRODUCTION
The beauty of skin and hair basically depends on individual’s health, diet, habits, job routine, climatic conditions and maintenance. Skin and hair disorders are the common ailments of all age groups because of chemical agents, toxins, microbes present in the atmosphere and also due to physical factors, malnutrition and environmental factors. One of the parts of beautification is the hair care, described in Ayurveda as ‘keshya karma’. Keshya karma is beneficial for hair and used for ‘vardhanam’ (promotion of hair growth) and ‘ranganam’ (dyeing of hair). The keya drugs with kesaranjan action include madayantika, bhringraj, nilika, bibhítaka, walnut, black catechu etc. Kesaranjan action of these drugs is also important to maintain aesthetic value of hair in keshavikar ‘paliya’ (premature graying) that has become very common nowadays. It is described in an Ayurvedic text that ideal hair should be soft, unctuous, having strong roots and should be black. According to the modern science, colour of the hair is due to the presence of pigments ‘melanin’ stored in the cortex of hair. Eumelanin, the dark pigment is responsible for hair shades from black to brown and phaeomelanin, lighter pigment is responsible for red and yellowish colors. Hair with no melanin pigments in cortex is completely white and with few pigments provides grey colour. Dissatisfaction with the colour of hair has led to the application of various types of natural and synthetic substances on hair. Synthetic hair dyes which are available in the market, uses combination of peroxide and ammonia which alters the structure of hair and damage it and also causes allergic reactions. Para-phenylenediamine (PPD), a key ingredient of many synthetic hair dyes is known to trigger allergic skin rashes in many people. It also causes dermatitis around lips, reddening and swelling of scalp and face etc. People using synthetic dyes are exposed to greater risk of developing urinary bladder cancer and non-Hodgkin’s lymphoma.

Vegetable dyes e.g. madayantika leaves, bhringraj etc are semi permanent dyes, used traditionally and believed to be safe and nontoxic. Plants have been used traditionally for their hair coloring, growth promoting and anti-aging properties. It has been found in the local market survey that the most of the marketed herbal formulations in India, though claim to be natural, safe and effective may actually contain the harmful synthetic agent, para-phenylenediamine (PPD), at 20-25% concentrations which is the main ingredient of commercial synthetic dyes (Table 1). As global scenario is now changing towards the use of safer, nontoxic natural product with traditional use, attempts have been made in the present investigation to develop herbal hair dye devoid of any chemical, containing few traditionally used herbs and modifiers; and compare it with marketed purely herbal hair dye. Herbo-mineral formulation containing different proportions of few herbs and loha / mandoor bhasma was prepared to get dark brown to black colour to hair as it is most favorite colour of Asian population. Number of hair creams, herbal oils containing extracts of mehendi, amla, bhringraj have been prepared, characterized and compared with marketed products for dyeing as well as growth enhancing activity. Classical formulations, Bhringaamalakaadi Taila, prepared with the fresh juice of

COMPARATIVE STUDY OF DYEING EFFICIENCY AND RETENTION CAPACITY OF HERBAL HAIR DYES
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ABSTRACT
In the present investigation, attempts were made to make a powder herbal hair dye that gives dark brown to black colour to hair, resembling natural hair colour with better dyeing effect and greater retention capacity on comparison with marketed herbal hair dye formulation. Most of the marketed herbal hair dye formulations contain para-phenylenediamine at 20-25% concentrations which is the main ingredient of commercial synthetic dyes. It is known to trigger allergic skin reactions in many people. Ayurvedic powder hair dye devoid of any synthetic agent was prepared in present research work and evaluated for dyeing efficiency. Different combinations of powdered leaves, fruits of plants like Madayantika, Bhringraj, Amla and Nilini were evaluated as hair dyes. The polyherbal hair dye compositions containing large proportion of Nilini, Mehandi and 5% Bhringraj and Amla, blended with 5% loha bhasma were prepared and evaluated for physicochemical parameters like particle size, colour, pH, angle of repose and bulk density. Ayurvedic polyherbal powder hair dye of present investigation is semi-permanent in nature and exhibit better dyeing efficiency than marketed herbal hair dye.

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Eclipta alba and Emblica officinalis; and Nilabhrringadi Taila prepared with the fresh juice of Nilika leaf, parts of Eclipta alba, and Emblica officinalis have been used to dye hair and promote hair growth since many years.

**MATERIALS AND METHODS**

Leaves of Madayantika (Lawsonia inermis) and Nilika (Indigofera tinctoria), Amla dried fruits (Phyllanthus emblica), whole herb of Bhringraj (Eclipta Alba) were procured, powdered and passed through the Sieve number 80. Loha bhasma was purchased from local market of India, Pune. All the samples were authenticated by expert botanist Dr. B. G. Kulkarni, scientist, botanical survey of India, Pune. Grey hair samples were collected from male and female volunteers.

**Formulation and Evaluation of Ayurvedic Herbal Hair Dyes**

Different grades (1-12) were assigned to colours ranging from jet black to blonde using an experimental color grade scale. White hair was used as a control for the in vitro study. Various dye formulations viz E, F, G, L, M and N were prepared using different ratios of Mehendi: Nili: Amla: Loha bhasma (Table 2). The herbal powders were weighed accurately, mixed geometrically followed by mixing in a polybag (Batch size: 100 g).

**Evaluation of Herbal Hair Dyes – In Vitro Studies**

The prepared hair dye formulations were studied for dyeing efficiency in vitro, on human white hair strands. Dyeing efficiency was determined in terms of colour grade and lasting capacity. Each herbal hair dye was applied to 0.5 gm of white human hair in vitro in a paste form. The dyed hair sample was washed with tap water after the period of 2 h; a second coat of dye was applied after 24 h of first application, kept for 2 h and then washed with tap water without aid of shampoo.

**Table 1: Few Herbal Hair Dye Products of Indian Market**

<table>
<thead>
<tr>
<th>Brand</th>
<th>Composition</th>
<th>Manufacturer / Marketed by</th>
</tr>
</thead>
<tbody>
<tr>
<td>Godrej Natural expert powder</td>
<td>Henna, Hibiscus, PPD – NLT 20% in powder form and NMT 2.5% on dilute form.</td>
<td>Godrej Hair Care Institute</td>
</tr>
<tr>
<td>Godrej Kali Mehendi</td>
<td>Amla, Shikakai, Reetha, Mehendi, PPD- NLT 25% in powder form and NMT 2.5% in</td>
<td>Godrej Consumer Products Ltd.</td>
</tr>
<tr>
<td>Indica Herbal hair color</td>
<td>Bhringraj, Amla, Methi, Henna and Hibiscus, Colorant and Softener PPD – NLT</td>
<td>CavinKare Private Ltd.</td>
</tr>
<tr>
<td>Garnier colour Natural</td>
<td>Hydrogen peroxide, Diaminophenol, Ammonia, Resorcinol and PPD (NMT 2.8%)</td>
<td>L’oreal</td>
</tr>
</tbody>
</table>

**Table 2: Colour retention capacity of Herbal hair dye formulations and marketed dye**

<table>
<thead>
<tr>
<th>Product code</th>
<th>Ratio of Mehendi: Nili: Amla : Loha</th>
<th>Colour grade (Day1)</th>
<th>Colour grade after number of shampoo washes</th>
</tr>
</thead>
<tbody>
<tr>
<td>E</td>
<td>4:4:0.5:0.5</td>
<td>2- Off black</td>
<td>2  2  3  4  5  5  5  6</td>
</tr>
<tr>
<td>F</td>
<td>5:3:0:2</td>
<td>4- Burgundy</td>
<td>4  4  5  6  6  6  7  7</td>
</tr>
<tr>
<td>G</td>
<td>4:4:0:2</td>
<td>3-Darkest brown</td>
<td>3  3  4  4  5  5  6  6</td>
</tr>
<tr>
<td>L</td>
<td>4:6:0:0</td>
<td>1- Jet black</td>
<td>1  1  2  3  4  4  5  6</td>
</tr>
<tr>
<td>M</td>
<td>6:4:0:0</td>
<td>4- Burgundy</td>
<td>4  5  7  8  8  8  8  8</td>
</tr>
<tr>
<td>N</td>
<td>5:5:0:0</td>
<td>2- Off black</td>
<td>2  3  3  4  4  4  5  5</td>
</tr>
<tr>
<td>Mktd</td>
<td>5:0:1:0.5</td>
<td>9- Fox red</td>
<td>9  9  10  11 11 12 12 12</td>
</tr>
</tbody>
</table>

**Table 3: Comparison of Colour retention capacity of Ayurvedic powder hair dye X and marketed dye M1**

<table>
<thead>
<tr>
<th>Product code</th>
<th>Ratio of Mehendi: Nili: Amla : Loha</th>
<th>Colour grade (Day1)</th>
<th>Colour grade after number of shampoo washes (in vitro study)</th>
<th>Retention property (No. of shampoos)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>4-4.5:0.5</td>
<td>2 – Off black</td>
<td>2  2  3  3  4  4  5  6  6</td>
<td>10-12</td>
</tr>
<tr>
<td>Mktd</td>
<td>5:0:1:0.5</td>
<td>9-Fox red</td>
<td>9  9  10  11 11 12 12 12</td>
<td>6-8</td>
</tr>
</tbody>
</table>

**Table 4: Physicochemical parameters of Ayurvedic test powder hair dye X and marketed product M1**

<table>
<thead>
<tr>
<th>Tests</th>
<th>Ayurvedic herbal hair dye X</th>
<th>Marketed herbal product M1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Fine powder</td>
<td>Fine powder</td>
</tr>
<tr>
<td>Colour</td>
<td>Light brown</td>
<td>Light brown</td>
</tr>
<tr>
<td>Odour</td>
<td>Characteristic</td>
<td>Characteristic (Madayantika)</td>
</tr>
<tr>
<td>pH</td>
<td>6.0</td>
<td>6.5</td>
</tr>
<tr>
<td>Particle size</td>
<td>80μ - 200 μ</td>
<td>80 μ - 160 μ</td>
</tr>
<tr>
<td>Angle of repose</td>
<td>24-28°</td>
<td>30 - 34°</td>
</tr>
<tr>
<td>Bulk density</td>
<td>0.388 g/cc</td>
<td>0.380 g/cc</td>
</tr>
<tr>
<td>Tapped density</td>
<td>0.495 g/cc</td>
<td>0.482 g/cc</td>
</tr>
</tbody>
</table>

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Figure 1: Dyeing effect of different powder herbal hair dye compositions on human white hair.

Figure 2: Photodocumentation of hair strands dyed using E, F, G, L, N and Marketed dye on 1st day and after 6 and 14 shampoo washes.

Figure 3: Comparative evaluation of dyeing effect of Ayurvedic herbal dye X with Marketed herbal hair dye M.

Figure 4: Photomicroscopic comparative evaluation of Ayurvedic and Marketed formulation dyed human hair strands.
The hair strands were observed on 1st day and alternate days after subsequent shampoo washes till 16 shampoo washes for following (Table 2, 3).

- Colour grade: Hair colour was graded as 1 to 12 by comparing with experimental colour grade scale. The observations were reported after 2, 4, 6, 8, 10, 12, 14 and 16 shampoo washes.

- Retention capacity / Colour lasting capacity: Retention capacity of herbal hair dye can be defined as ability of hair dye to retain or last particular hair colour on white /blonde human hair for definite period of time. In present investigation, the hair was washed with mild shampoo on alternate days using tap water till 16 shampoo washes (32 days) and retention capacity was determined in terms of number of shampoo washes that a colour can withstand.

- Photodocumentation: The white hair dyed with different compositions viz. E, F, G, L, M, N and marketed herbal dye were photographed using Sony digital camera (14.1 mega pixels), keeping the distance between sample and camera constant and other settings of camera e.g. mode, brightness, light effects were also kept constant (Figure 1). The hair strands were also photographed after 6 and 14 shampoo washes (Figure 2).

**Formulation of Ayurvedic Hair Dye X**

Ayurvedic herbal hair dye X was prepared based on findings from compositions E, F, G, L, M, N in a large batch size (500 gm) to get better colour. The formulation X was standardized for physicochemical parameters and compared with marketed herbal hair dye M1 (Table 3, 4) for dyeing efficiency and retention capacity (Figure 3).

The photomicrographs of same hair strand samples were taken using compound electron microscope under 10X magnification, using 4x optical zoom system of camera, and penetration efficiency of both the dyes was observed in the medulla and cortex region of hair strands (Figure 4).

**RESULTS AND DISCUSSION**

Darker colour shades 1-3 were not obtained using compositions F, M and so they were not selected. Compositions E, G, L, N were found to produce off black to dark brown colour initially (grades1-3) and fades gradually to grade 5.6 (chestnut – medium brown) after 14 to 16 shampoo washes (Figure 1 and 2, Table 2). Dyes ‘E’ and ‘N’ produce grades 3 and 4 while dye ‘L’ showed grade 2 and 3 after 6 and 8 washes and grade 3 (darkest brown) till 8 shampoo washes. Powder composition X using (2:2.25) ratio of madayanti : nili ; (1:1) ratio of amla : loha bhasma and additional ingredient bhringraj powder was prepared and characterized for dyeing properties after 2 times application on white hair in order to get better results viz: darker colour (grade1/2) and better retention (10-12 shampoo washes).

**Comparison of Test Powder Hair Dye X with Marketed Dye M1- In Vitro Evaluation**

Dye X was compared with marketed dye M1 for colour grade, retention capacity and photographs. The formulation X dyed the hair strands off black when tested in vitro and showed retention capacity of at least 10-12 shampoo washes (Table 3, Figure 3). Test dye X fade to grade 4 (burgundy) after 10 washes, however marketed dye M1 gave fox red colour (grade 9) which fade to grade 11 (golden blonde) and 12 (pale blonde) after 10-12 shampoo washes. The retention capacity of marketed dye M1 was found to be only 6-8 shampoo washes, much lesser than test dye X. Comparison of microscopic photograph (Figure 4) further confirmed the darker grade (off black) produced by Ayurvedic dye X in the medulla region as compared to marketed dye M1. Thus the desired off black colour can be obtained by selecting the proper proportions of mehendi, nilini, loha bhasma, amla and maka. Both the formulations were compared with respect to various physicochemical properties (Table 4).

The increased dyeing effect and better retention capacity of developed ayurvedic dye X may be attributed to following. The various constituents present in these herbs such as coloring principles lawsone (mehendi), indigotin (nili), tannins and minerals in combination with metallic dye loha bhasma produce desired burgundy to off black colour.

- Dyeing action of madayanti may be attributed to reaction of lawsone (thiol group) with keratin of hair. It was also suggested that dyeing action is because of acceleration of blood circulation, activation of dermal papilla and increased nutrition to the hair follicle.

- Indigofera, one of the oldest and widely used coloring agents in the world contains ‘Indigothin’ that gives violet colour to hair. Kesharanjan action of nili results into krishnakesha and it is described in Nighantu period as neelkeshi and krishnayavanjanakesi.

- The mechanism of action of maka is not clear but it is considered to blacken the hair and nourish them.

- Dried fruits of amla have been used in different shampoo and oil preparations, for promotion of hair growth and prevention of hair greying- classic sign of pitta dosha. Amla fruit is tridoshashamaka, especially pittashamaka and is effective against manifestations of pitta on hair. Amla alleviates pitta dosha due to its sheeta and madhura properties and thereby promotes pigmentation of hair.

- Loha bhasma is a microfine powder of iron oxide containing Fe, Fe$_2$O$_3$, Fe$_3$O$_4$ and interaction of iron oxide with fine amla powder (ascorbic acid) produce fused black particles (chelates) capable of dyeing hair. Thus modifiers amla and loha bhasma in 1:1 ratio in a formulation enhance penetration of black particles as well as lawsone and indigotin, deep into the medulla region, thereby increasing colour intensity and retention property.

- Further tannins present in large amounts in amla create affinity between adhesive dyes and hair. Microscopic evaluation facilitates the observation of colour grade, intensity and penetration of a dye. Dyeing efficiency of ayurvedic dye X and marketed dye M1 was also compared using photomicrographs of X and M1 dyed hair strands. Grade 2 colour and increased penetration was observed in case of Ayurvedic dye X when compared...
with Marketed herbal M1 dye. Thus the developed polyherbal dye formulation has number of significances.

- The desired off black hair colour can be obtained by selecting the proper proportions of methendi, nilini, loha bhasma, amla and maka.
- Use of amla and maka in a developed formulation helps in promotion of hair growth along with blackening effect. It can also provide other effects such as conditioning and calming effect.
- As the hair dye formulation is prepared from authenticated herbs and is free from any synthetic harmful ingredient such as PPD, it will exhibit minimal toxicity as compared to existing herbal and synthetic marketed dyes.
- The composition and method of preparation is very simple, highly economical and simple to use.

**CONCLUSION**

The developed powder herbal hair dye is promising natural hair colorant and recommended for use as dark brown to black dye. Ayurvedic polyherbal powder hair dye of present investigation is semi permanent in nature and exhibit better dyeing efficiency than marketed herbal hair dye. The developed ayurvedic hair dye formulation is prepared from purely natural substances, free from chemicals, so considered to be non irritating, non toxic and devoid of any side effects such as hair fall, dandruff production etc. Further studies should be performed to evaluate dyeing efficiency and toxicity in human volunteers.

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