

**PREPARATION, EVALUATION AND HAIR DYEING ACTIVITY OF  
HERBAL HAIR OIL AND COMPARISON WITH MARKETING DYE****Phadatare Suvarna P<sup>1\*</sup>, Nesari Tanuja N<sup>2\*</sup>, Pokharkar Deepak<sup>3</sup>, Pingle R.P<sup>1</sup>**

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Article Received on  
11 June 2015,

Revised on 02 July 2015,  
Accepted on 23 July 2015

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**ABSTRACT**

The usage of herbal cosmetics has been increased to many folds in personal care system and there is a great demand for herbal cosmetics that offer multifunctional effects. Most of the marketed herbal hair dye formulations contain para-phenylenediamine at 20-25% concentrations which is the main ingredient of commercial synthetic dyes. In the present investigation, attempts were made to make an oil herbal hair dye that gives dark brown to black colour to hair, resembling natural hair colour. The polyherbal hair oil was prepared using amla, mehendi, maka, nili and evaluated for dyeing effect on white human hair strands in vitro. The mixture of crude drugs, fruits of *Embelica officinalis*, leaves of *Lawsonia innermis*, *Indigofera tinctoria* and whole plant of *Eclipta alba* were processed in varying concentrations using Ayurvedic method of oil preparation viz. Taila Pak Vidhi and

evaluated for dyeing efficiency. The oil was also characterized for proximate analysis including moisture content, total ash, acid insoluble ash, water soluble ash, water insoluble ash, sulphated ash. Excellent results were obtained with developed formulation and the time (in days) required for hair darkening effect was found similar with marketed oil.

**KEYWORDS:** Herbal formulation, Hair oil, Hair dyeing, Nilini, Bhringraj, Mehendi, Amla.

## INTRODUCTION

A global scenario is changing towards the use of safer, nontoxic and natural products with traditional use. Herbal hair preparations are used in various disorders such as alopecia, dandruff, greying, premature greying and head lice etc.<sup>[1,2]</sup> Concealing grey hair or modifying natural hair colour has been increased to great extent and various types of natural and synthetic dyes are used to give colour to hair. The natural 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 pheomelanin, lighter pigment, for red and yellowish colors. Hair with no melanin pigments in cortex is completely white and with few pigments is still grey.<sup>[1]</sup>

Hair care products are categorized into two main category, hair tonics and hair grooming aids.<sup>[2,3]</sup> Herbal hair oils are basically the extracts of medicinal plants in an oil base and can be used as tonic as well as grooming aid. Synthetic oxidative hair dyes available in the market contain combination of peroxide and ammonia which damage hair and causes allergic reactions. Para-phenylenediamine (PPD), a key ingredient of many synthetic hair dyes triggers allergic skin rashes, dermatitis around lips, and reddening of scalp and face etc in many people.<sup>[4]</sup> Further the people using synthetic dyes are exposed to greater risk of developing urinary bladder cancer and non-Hodgkin's lymphoma.<sup>[5-7]</sup>

A number of herbs have been used traditionally for their hair coloring, growth promoting and anti aging properties. A few of them are amla, neem, maka, heena, lemon, brahmi, tulsi, shikakai, reetha, nutmeg, thyme etc.<sup>[3,8]</sup> Vegetable dyes eg. Mehendi leaves, Bhringraj (maka) etc are used traditionally and believed to be safe and nontoxic. Most of the marketed herbal oil formulations intended for colouring of hair, contain the harmful synthetic agent, para-phenylenediamine (PPD), at 20-25% concentrations which is the main ingredient of commercial synthetic dyes (Table 1). Attempts have been made in the present investigation to develop Herbal hair oil dye devoid of PPD, containing different proportions of few traditionally used herbs mainly *Lawsonia inermis* and *Indigofera tinctoria* and to evaluate its hair dyeing property in comparison with marketed herbal oil dye.

**Table 1: Few marketed herbal formulations**

Sr.	Brand and type	Composition	Mfger / Marketed by	Claims and shelf life
1	Godrej Kesh Kala Oil based hair dye	Coconut & natural extracts of Mehendi, Amla, Shikakai and Bhringaraj. PPD NMT 4% in dye ready for use	Mkted by: Godrej Consumer Products Ltd.	For healthy Black Hair, Shelf life : 2yrs
2	Vasmol Kesh Kala Emulsion type hair dye	PPD NMT 4 %	Mkted by: Hygienic Research Institute Ltd.	No ammonia, no peroxide, Shelf life : 2yrs
3	Anoop Hair Oil	Milk and coconut oil base, Extracts of I. tinctoria, E. alba, E. Officinalis, G.glabra C. haliccabum,	Mfger: Arshik Herbal Remedies (I), Mkted by: Godrej Consumer Products Ltd.	100 % herbal. Shelf life : 3yrs
4.	Saini Herbal Oil	Amalaki, Shikakai, Jajoba oil, Bhringraj, Daruhaldi, Karpoor, Til oil, Liq paraffin	Mfger: Saini hair Products Pvt ltd.	Non sticky, Shelf life : 3 yrs
5.	Krishnakesha Taila	Centella asiatica ; Eclipta alba , lawsoniainermis	Agom Aushadhalaya Pvt Ltd.	Stops hair falling and greying Shelf life : 2 yrs

Number of herbal hair preparations containing extracts of mehendi, amla, bhringraj have been prepared, characterized and compared with marketed products for dyeing as well as growth enhancing activity.<sup>[2,9,10]</sup> Classical examples of oil formulations are Bhringaamalakaadi Taila (fresh juice of Eclipta alba and Emblica officinalis) and Nilibhringaadi Taila (fresh juice of Indigo leaf, Eclipta alba, and Emblica officinalis).<sup>[11]</sup>

Selection of Mehendi and Nili in present study was based on the fact that leaves of these plants produce orange red and purple colour respectively,<sup>[12,13]</sup> and a proper combination of both should provide burgundy to black colour to hair. The formulation of Mahanila taila contains both, madayanti and nili.<sup>[14]</sup> Oil formulations are easy to apply, mostly stable at room temperature and regularly used by many people for its softening, conditioning and calming effects. The market for hair dye products is also expanding to great extent in India as well as abroad. Hair dyeing is now a multibillion dollar industry that involves the use of both plant-derived and synthetic dyes.<sup>[15]</sup>

## 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 from local market of Pune. All the samples were authenticated by expert botanist, powdered in

mixer and sifted through the sieve number 80. Grey hair samples were collected from male and female volunteers. Drugs were subjected to pharmacognostic studies for confirmation.

### Preparation of polyherbal oil hair dye by tail pak vidhi

Formulation of Oil Hair Dye (S<sub>2</sub>) as per table2 was prepared. The ratio of Oil : Herb : water (4: 2 : 16 ) was selected for preparation. Extract of Maka (100 ml) was prepared by boiling coarse maka powder in 16 times of water in a S.S. vessel, reduced to ¼<sup>th</sup> volume and strained using muslin cloth (Kwatha dravya). The mixture of oil: water in 1:2 ratio was heated to temperature 60 °C in S.S. vessel. Nili and Madayantika powder was passed through sieve no 85 and transferred to grinder for wet mixing with distilled water (1Lit.) to prepare homogenous blend (Kalka). Kalka was then added to oil : water mixture with mechanical stirring. Stirring speed was maintained at 200 rpm. Kwatha dravya of maka, amla powder and remaining quantity of water was then added to a vessel with continuous stirring. The mixture was heated in S.S. Vessel for 3 hours with continuous stirring, maintaining temperature at 60° C during 1<sup>st</sup> hr of heating, and at 70°C- 80°C in next 2 hours. The mixture was allowed to stand overnight. Heating was started next day with stirring and kalka was checked for in process Quality Control (IPQC) tests to ensure completion of process. Heating was stopped at the appearance of froth over oil and kalka was checked for formation of varti. Varti was tested for absence of moisture (Table 3, Fig1).

The final mixture was filtered while hot (about 70° C) through muslin cloth and allowed to cool. The oil was stored in tightly closed, opaque bulk container to protect from light and moisture. The oil was transferred to 60 ml amber colored glass bottles and sealed.

**Table 2: Formulation of poly herbal oil hair dye S<sub>2</sub>**

Sr. No	Ingredients	Quantity
1.	Mehendi : Nilika	2: 2.25
2.	Amla powder	0.25
3.	Maka	0.25
4.	Distilled Water : oil	40:10
5.	Coconut oil+ sesame oil (1:1)	10

**Table3: In process standardization of herbal oil hair dye S<sub>2</sub> ( Ayurvedic method )**

Sr. No.	Tests	Observation
1.	Khar paka laksana- paste / kalka roll between fingers.	Varti formed breaks into pieces, waxy in nature.
2.	Test for absence of moisture- Exposure of varti to flame	No cracking sound indicating absence of moisture

3.	Processed oil + fire	Burns without noise
4.	Test for well processed oil	Appearance of foam or bubbles on surface of oil
5.	Colour and odour	Very dark brown with characteristic pleasant odour, that of herbs used.



Mechanical mixing of drug extracts  
and paste in Taila Pak vidhi



Froth formation – completion of  
Process-Taila pak vidhi

**Fig 1: Tail Pak Vidhi for preparation of Natural Herbal Oil Hair Dye S2.**

### Evaluation of herbal oil hair dye – in vitro studies

The developed formulation oil hair dye S<sub>2</sub> was evaluated using standard methods of general characterization, including appearance, colour, odour, specific gravity, pH, Refractive index, acid value, saponification value etc. and compared with poly herbal marketed oil hair dye. (Table 4, 5).

For evaluation of dyeing effect, nominal scale (grades 1-12) is used to grade different hair colours ranging from jet black to blonde and referred as 'experimental colour grade scale' in this study. Reduction in colour grade indicates appearance of darker hair colour and helps in comparison of darkening property (table 5) of dyes in the present study. Darkening property is the qualitative parameter indicating the ability of the hair dye to darken the hair or to increase colour intensity. It is determined in terms of number of applications that an oil hair dye requires to get a particular hair colour. White hair was used as a control for the *in vitro* study.

Each herbal hair dye was applied to 0.5 gm of white human hair in vitro and applications were repeated every day, without washing hair. Hair colour was graded between 1 to 12 using colour grade scale. The observations were reported after 2, 4, 6, 8, 10 and 12 applications. Colour grade produced by a test dye after definite number of applications on human white hair strands was compared with marketed dye (Table 5). The white hair dyed with test dye S<sub>2</sub>

and marketed dye M2 were photographed using Sony digital camera (14.1 mega pixels), keeping the distance between sample and camera constant and other settings of camera eg. mode, brightness, light effects were also kept constant (Fig.2). The two hair dyes were also compared microscopically for evaluation of dyeing effect. 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 (Fig 3).

## RESULTS AND DISCUSSION

Coconut oil and tila oil (1:1 ratio) was selected as vehicle for oil hair dye preparation. Sesame oil is preferred because of its good stability due to natural antioxidants sesamin and sesamol.<sup>[16-18]</sup> Coconut oil is most widely used hair oil because of its nutritional benefits. It enhances strength of hair and also prevents dryness of hair. Scientific method 'Tail Pak Vidhi' given in Ayurvedic Pharmacopoeia of India was followed for preparation of medicated oil hair dye<sup>[19]</sup> using ratio 2 : 4 : 16 of Herbs , Oil and Water. In process quality control tests were found to comply and given in table 3.

Developed hair Formulation was compared with selected marketed formulation for different physicochemical tests and results are given in table 4. The test dye S2 was more dark coloured oil with pH > 6 and also it is more viscous than marketed oil. Both dyes S2 and M2 were applied to hair *in vitro* and colour grade was observed after 2, 4, 6, .....10, 12 applications. Marketed oil imparted grade 10 colour to hair sample initially and then darkened to grade 6 ( medium brown) after 12 applications. Test herbal dye S2 showed darker grade 8 (medium ash brown) initially and changed gradually to grade 5 (chestnut brown) after 12 applications (table 5).

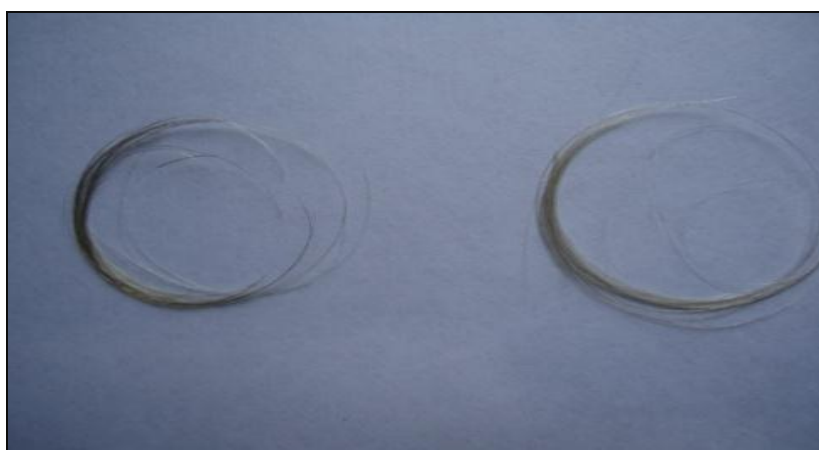
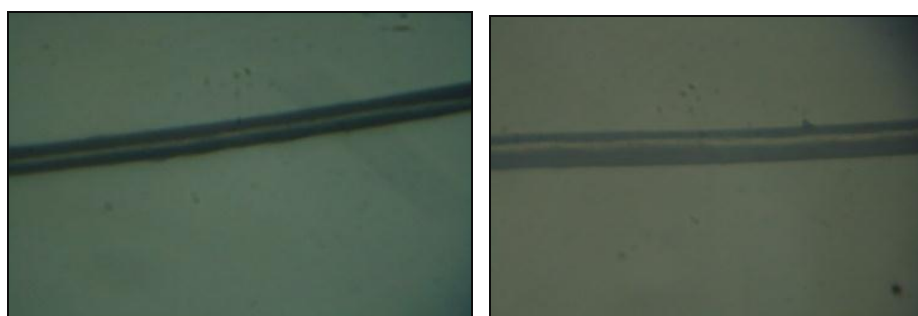
**Table 4: Comparison between Ayurvedic oil hair dye S<sub>2</sub> and Marketed product M<sub>2</sub>**

Sr. No.	Physical Tests	Herbal oil hair dye S <sub>2</sub>	Marketed herbal oil product M <sub>2</sub>
1.	Colour	Dark brown oil	Brown oil
2.	Odour	Characteristic	Characteristic
3.	pH	6.2	5.6
4.	Weight / ml	0.985 g/ml	0.94
5.	Refractive Index (35°C)	1.465	1.35
6.	Viscosity	32 cps	28 cps
7.	Acid value	2.56	2.47
8.	Saponification value	235.5	232.9
9.	Moisture content	0.045%	0.051%



**Table 5: Comparison of Dyeing Effect of Oil Hair Dye S<sub>2</sub> and Marketed product M<sub>2</sub>**

Sr. No.	Hair Oils	No. of Applications					
		02	04	06	08	10	12
1.	Lab Hair Oil S2	Medium ash brown (Grade 8)	(8)	Medium golden brown (6)	(6)	Chestnut brown (5)	(5)
2.	Mktd hair Oil M2	Strawberry Blonde (10)	(10)	(9)	(9)	Fox red (9)	Medium golden brown (6)

**Test dye S2****Mktd Dye M2****Fig 2: Comparative dyeing effect of Natural Oil herbal dye S2 with Marketed Oil hair dye M2 on white human hair strands- in vitro study. (After 8 applications)****Test oil S2****Mktd oil M2****Fig 3 : Microphotographs of dyed hair for comparison of dyeing effect of test herbal dye S2 with marketed dye M2. (After 8 applications)**

Photographs of hair strands after 8 number of applications of test dye S2 and marketed dye M2 are shown in fig. 2. Test dye S2 resulted in colour grade 6 (medium golden brown) and M2 resulted in grade 9 (fox red) after continuous 8 applications of oil. Dyeing effect of both the dyes was compared microscopically after 8 applications and revealed that penetrability of

both dyes was comparable, however dark colour grade was obtained with test oil dye S2 (Fig 3).

Thus the present study revealed that the various coloring principles such as lawsone (*L. innermis*), indigotin (*I. tinctoria*) in combination with tannins and minerals of the herbs impart colour to white hair. Repeated applications of oil type hair dyes, both sample and marketed dyes, gradually give darker colour (lower grades) to white hair. However, developed formulation results in darker hair colour as compared to marketed oil dye after same number of applications. As shown in table 5, chestnut brown hair colour (grade5) was obtained with test dye S2 after 12 applications on white hair in vitro. Marketed dye M2 imparted medium golden brown (grade 6) hair colour after 12 applications, however same grade 6 hair colour was obtained just after 6-8 number of applications using dye S2.

Thus both oil dyes S2 and M2 imparted brown shades but were unsuccessful in imparting desired burgundy (grade 4) to off black colour (grade 2) to hair in vitro. However the developed dye S2 showed better dyeing effect viz. darker chestnut brown colour grade against medium golden brown colour by M2 after 12 applications; and imparted medium golden brown colour in lesser number of applications as compared to M2. Further the use of amla and maka in composition may help in nourishment and promotion of hair growth along with blackening effect.

Dyeing action of oil formulation may be attributed to reaction of 'Lawsone' (thiol group) of mehendi (orange red pigments) and 'Indigotin' of Nili (violet) with keratin of hair.<sup>[12,13,20,21]</sup> Penetration of lawsone and indigotin, deep into the medulla region with repeated applications results in darker colour grade in case of test dye S2. Further it was suggested that tannins present in large amounts in amla create affinity between adjective dyes and hair.<sup>[3]</sup> Microscopic evaluation facilitates the observation of colour grade and penetration. Comparison of photomicrographs (fig 3) of dyed hair indicate that penetrability of both dyes was comparable.

As the hair dye formulation is prepared from authenticated herbs and 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 of developed oil herbal hair dye is simple and formulation is stable at room temperature. Formulation provides added advantages such as ease of application, no rinsing of hair



required after application, no waiting duration of 2 h or more, no staining of skin and hands in contrast to powder herbal and synthetic dyes. Further intensity of hair colour will increase day by day with regular use of oil hair dye. Use of oil type hair dye may also result in reduction of hair damage and improved manageability.

## CONCLUSIONS

Developed oil hair dye prepared by Taila pak vidhi, holds the promise of potent natural herbal hair dye to get dark brown to burgundy hair colour and provides a better alternative to existing purely herbal hair dyes. Further modifications in composition such as use of more herbs viz. Bixa seeds, Tea leaves etc. may result in oil hair dye imparting desired burgundy brown to black colour as it is assumed that it is the most preferred hair colour of Asian population. The developed oil hair dye may provide multifunctional effects such as softening and conditioning effect, promotion of growth and density of hair, calming effect etc., because of use of amla and maka in composition, along with darkening of hair. This could satisfy the need of the population of all age groups.

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