



Development and Evaluation of Polyherbal Hair Dye Formulation

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Abstract

Background and Objectives: Hair care products including hair dye play an important role in the maintenance and enhancement of appearance and overall wellbeing of the hair. Most of the hair dyes available in the market contain harmful chemicals that may cause allergic reactions or irritation of the scalp. The objective of this study focuses on the formulation and evaluation of hair dye as creams and gels with different shades by using herbs. **Methodology:** The collected plants were authenticated, and shade dried. Then, extractions were done using water, ethanol, and methanol. Preliminary testing was conducted to obtain burgundy, brown and black by manipulating the ratios. Later, almond oil, coconut oil, beeswax and Carbopol-934 gel were added to ease the penetration. Finally, accelerated stability studies were done to check the physical and microbial stability of the product. **Results:** Eight different shades of hair dye were formulated in gel and cream form. Among them, gel formulations F5 and F6 as well as the cream formulations F1, F2, F3, F4, F7 and F8 demonstrated good staining and better colour intensity. The accelerated stability studies of F1 to F8 revealed that there is no change in the physical evaluation and no evidence of microbial contamination. **Conclusion:** The output of this research is suitable for all races and age groups because it is not a permanent hair dye that might have a restriction for some groups of races. As such, this hair dye is fully formulated with natural sources by considering the reduction of severe side effects to the consumers.

Keywords: Creams, Gels, Herbal Extracts, Herbal Hair Dye, Human Hair

1. Introduction

Hair acts as an intrinsic part of the body by reflecting the image, and it can easily change someone's personality without resorting to surgical procedures. Healthy hair is considered an asset in this generation because people tend to spend more time on their hair to look perfect and lustrous. Therefore, fancy cosmetics and techniques have been used since ancient times to change the appearance of hair. To meet the demand, the beauty industry continues to establish reliable goods that are used to treat healthy hair and scalp diseases together and many cosmetic companies want to enter

the hair colour or hair dye industry. Regarding the hair dye, the dyes exaggerate the hair by bleaching or colouring it for a short time or longer time, depending entirely on the composition of a dye that has oxidative or non-oxidative¹.

Currently, the whole world is turning back to ancient days when only herbs were used for multipurpose functions. In this 21st century, all are going towards the usage of herbal products and trying to adopt a more natural way of life. People prefer natural food, herbal medicine and also natural curing practices for a healthy life. All this happened due to the excessive use of synthetic products, chemicals, synthetic dyes, and their

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derived products in the last one and a half-century, and the usage of these kinds of synthetic products causes human health hazards with side effects that may lead to many kinds of diseases compared to that days².

1.1 Classification of Hair Dye

There are four most common classifications of hair colours: they are temporary non-oxidative, semi-permanent hair dye, permanent hair dye and milder hair dye. Firstly, temporary non-oxidative hair dye is effective for brightening the colour of the original hair, adding a tint or improving the colour of the hair. It can be rinsed off through water and rarely lasts until the next hair. Temporary hair colour is available in various forms including rinses, shampoos, gels, sprays, and foams³. 'Silver shampoo' is an example of a temporary dye used to give a silver hair look to women with all-grey hair¹.

For semi-permanent hair colour which can penetrate the hair shaft has a smaller size of molecules compared to temporary dyes. That is why this hair dye lasts a couple of weeks or four to six washes. This type of dye contains very low levels of developers, peroxide or ammonia. Moreover, it contains toxic substances such as p-phenylenediamine or other agents³. Nowadays a few items like lotions, shampoos, mousses, and emulsions are currently available on the market for hair products⁴.

Next, permanent hair colouring is generally used when this category offers better effectiveness than permanent dyeing, which has shampoo resistance as well as other external factors. Most of such hair colours of this type are selling well, are available in many shades and cover all the white hair strands. Due to a combination of the oxidizing agents with those of the ammonia hydroxide, it is also possible to have dark and light natural hair colour. Permanent colours are everlasting and don't get washed out even though they may become paler³. Permanent hair colours such as liquids, gels and creams are obtainable⁴.

Lastly, there are middle hair dyes which are milder on the hair than permanent colours since they do not contain ammonia and also have a lower hydrogen peroxide concentration compared to permanent hair colours. Thus, scalp irritation and hair shaft damage are lowered. They are beneficial in hiding grey hair, yet do not lighten the colour of the hair¹.

Herbal hair dye is not easily available in the market in powder form which can give different shades.

Moreover, the hair dyes available in the market contain harsh chemicals which can damage the scalp and cause dryness to the hair. Therefore, the objective of this study is to formulate and evaluate hair dye with different shades by using natural sources. And the hypothesis of this study is, 'The formulated hair dye will stain the hair with an appropriate colour'.

2. Materials and Methods

2.1 Materials

The plants mentioned were collected from Klang Valley, Malaysia. Seeds of fenugreek (*Trigonella foenum-graecum* L.), the fruit of noni (*Morinda citrifolia* L.), the whole plant of Bhringraj (*Eclipta alba* L.), leaves of Henna (*Lawsonia inermis*), petals of hibiscus (*Rosa-sinensis* Linn), the bark of cinnamon (*Cinnamomum zeylanicum*), and leaves of pandan (*Pandanus amaryllifolius*), methanol (Sigma-Aldrich, Malaysia), ethanol (Sigma-Aldrich, Malaysia), and distilled water.

2.2 Collection and Drying of Plants

Required parts of the plants were collected from Klang Valley and the identification and authentication were done at the Institute of Bioscience University Putra Malaya (UPM), Malaysia. All plants collected were cut into small pieces and spread on a tray. Plants are then dried under the sun until no significant presence of moisture, and they can easily be broken by crushing using a hand. The hair sample was collected at a salon in Bukit Tinggi, Klang, Selangor. Pure cotton was used for preliminary testing⁵.

2.3 Size Reduction of Dried Plants

All dried plants were crushed using the cone mixer or/and mortar pestle accordingly. The crushed product was then further sieved using the electronic sieve shaker to ensure the uniformity of size. Dried plants were weighed using the analytical balance before being stored in the bottle⁶.

2.4 Extraction and Evaporation of Herbs

2.4.1 Aqueous Extraction Method

An aqueous solution of the dried parts which was 3.3gm in 100ml distilled water was prepared. The beaker was heated at 95°C on the hot plate for 2 hours with the mechanical stirrer in it. Then, the extraction was

filtered, and the volume obtained from the extraction was measured².

2.4.2 Aqueous-alcohol Extraction Method

The finely crushed dried flowers were taken in three different beakers containing 95% ethanol or 96% methanol. The beaker was heated over a hot plate with a magnetic stirrer in it for 4 hours at 68°C and was filtered out. The raw material was retained in a thimble of the rotary extractor and the solvent was poured into a round bottom flask and the condenser with a high flow rate of water was fitted over it. The extraction was carried out until the desired extraction was obtained and the volume of the solution was measured².

2.4.3 Specific Extraction Method

2.4.3.1 Pandan Extraction

Dried pandan leaves mixed with 95% ethanol which was kept overnight and stored in a cold dark place with a temperature of 20°C. After that, 0.1% of hydrochloric acid (HCl) was mixed with that extraction and was left at room temperature for 2 to 3 hours. The mixture was then filtered and the remaining solids were washed with citric acid until a clear solution was formed⁷.

2.4.3.2 Cinnamon Extraction

Dried cinnamonbarks were mixed with 96% methanol and were kept in a cold dark place for 3 days. After 3 days, the extraction was filtered and was exposed to the water bath for 30 minutes⁸.

2.4.3.3 Hibiscus Extraction

The dried hibiscus was mixed with 96% methanol and was kept overnight and stored in a cold dark place with a temperature of 20°C. After that, 0.1% of HCl was added to the extraction and was left at room temperature for 2 to 3 hours. The mixture then was filtered and the remaining solids were washed with citric acid until a clear solution was formed⁹.

2.4.4 Evaporation Process

A hot air oven was used to remove the moisture from the samples for the drying of herbal plants. All the extractions were oven-dried at 50°C until it left a very thick consistency of extraction¹⁰.

2.4.5 Preliminary Studies

The obtained residue was used to test for burgundy, brown and black. Two tests were conducted under preliminary testing. Firstly, identification of colour stained was done on the cotton for all three colours which are burgundy, brown, and black. It was to test the intensity of the stain on the cotton. Followed by, individual identification of cotton to check the staining of each plant for a particular colour¹.

2.4.5.1 Selection Ratio of Herbs for Burgundy Stain

For burgundy colour, dried hibiscus, cinnamon and fenugreek were used. The mixture was prepared by using a ratio of 1. All three dried herbs were mixed in a petri dish with two drops of distilled water. The mixture then has been pasted onto the cotton and left for 30 minutes before washing out¹¹. Table 1 shows the ratio taken for each plant to produce burgundy colour.

Table 1. Ratio for the burgundy colour

Hibiscus (Specific)	Cinnamon (Specific)	Fenugreek (Ethanol)
1	1	1
1	2	1
2	1	1

2.4.5.2 Selection Ratio of Herbs for Brown Stain

For brown colour, dried henna, cinnamon and fenugreek were used. The mixture was prepared with a ratio of one. All three dried herbs were mixed in a petri dish with two drops of distilled water. The mixture then was pasted onto the cotton and was left for 30 minutes before being washed out of the cotton¹¹. Table 2 shows the ratio taken for each plant to produce a brown colour.

Table 2. Ratio for the brown colour

Cinnamon (Specific)	Henna (Specific)	Fenugreek (Ethanol)
1	1	1
1	2	1
2	1	1
2	2	1

2.4.5.3 Selection Ratio of Herbs for Black Stain

For black colour, dried eclipta, noni, hibiscus and fenugreek were used. The mixture is in a ratio of one for all the plants. All three dried herbs were mixed in a petri dish with two drops of distilled water. The mixture then was pasted onto the cotton and was left for 30 minutes before being washed out of the cotton¹¹. Table 3 shows the ratio taken for each plant to produce a black colour.

Table 3. Ratio for black colour

Noni (Ethanol)	Eclipta (Specific)	Hibiscus (Ethanol)	Fenugreek (Ethanol)
1	1	1	1
1	2	1	1
1	1	2	1
2	1	1	1
1	2	2	1
2	2	1	1
2	1	2	1

2.4.5.4 Selection Ratio of Herbs for Green Stain

For the green colour, dried pandan leaves and fenugreek seeds were used. The two dried herbs were mixed in a petri dish with two drops of distilled water. The mixture then was pasted onto the cotton and was left for 30 minutes before being washed out of the cotton¹². Table 4 shows the ratio taken for each plant to produce a green colour.

Table 4. Ratio for the green colour

Pandan (Specific)	Fenugreek (Ethanol)
1	1
2	1

2.4.6 Formulation of Hair Dye

An accurately weighed amount of each herb was added into a small petri dish according to the ratio determined earlier. The mixture of all herbs to form one group of colours was mixed with gel or beeswax

that was prepared earlier. The formulation was applied to the white hair collected and after a while, it was washed thoroughly with running water.

The gel was formulated by using Carbopol 934, which was weighed according to the ratio scale of six and added into a centrifuge tube in which 10 distilled water was mixed. The vortex mixer was turned on to vortex vigorously until it formed a gel-like consistency. Steps were repeated for each formulation that consisted of gel formation.

For the formation of cream, beeswax was weighed according to the ratio scale of six and was added to a petri dish. Then, it melted over the hot plate. Steps were repeated for each cream formulation that consisted of beeswax.

2.4.7 Evaluation Testing for the Finished Product

Evaluation testing for the finished product was done using cotton fibre and human white strands to determine the dyeing efficiency in terms of colour intensity¹³.

2.4.7.1 Study on Dyeing Effect

The formulated dyes were kept side by side for 1 hour for imbibition and then the cotton samples were kept for 1 hour to 2 hours for all the different shades of cotton dye formulations for their dyeing effect¹⁴.

2.4.7.2 Evaluation of Herbal Hair Dye Using Human Hair

The prepared formulation was studied for dyeing efficiency, which will be on human white strands. Dyeing efficiency was determined in terms of colour grade. Accurately, 0.33gm of herbal hair dye was applied to human white strands. Then, the dyed white hair was washed with tap water after 2 hours. Followed by the second coating of the dye that needs to be applied after 24 hours of the first application and was kept for 2 hours and was washed again with tap water without the aid of shampoo¹³.

2.4.8 Accelerated Stability Studies

Stability testing is usually tested with the prepared formulation by storing it at different temperature conditions for the period according to the individual's study¹⁵. After evaluation studies of sixteen formulations only eight formulations that passed the tests were selected

and tested for stability studies. The finalized eight formulations of hair dye were individually kept in a 30g small amber jar and were sealed with parafilm all over the container to prevent contamination. The product was kept in the humidity chamber with a temperature of 40°C and humidity at 75% for three months¹⁶.

2.4.8.1 Physical Evaluation

The packed glass vials of formulation were stored at different temperature conditions and were evaluated for the physical parameters like colour, odour, texture, and smoothness¹⁵.

2.4.8.2 Microbial Contamination

The packed glass vials of formulation were stored at different temperature conditions and were evaluated for any colour change. When the products are contaminated, the microorganisms can cause changes like discolouration, and texture alteration¹⁷.

3. Results and Discussion

3.1 Preliminary Studies

3.1.1 Individual Stains

Various colours of dye were formulated based on the combination of herbs as mentioned in Table 5.

Table 5. Plants used for each individual stain

Stains	Herbs
Burgundy	Cinnamon: hibiscus: fenugreek
Brown	Cinnamon: henna: fenugreek
Black	Eclipta: noni: hibiscus: fenugreek
Pandan	Pandan
Fenugreek	Fenugreek

3.1.2 Combination of Colour

A combination ratio was used for a particular colour stain on the cotton before and after wash indicating the intensity of the colour. The combination of colours is classified according to the colour stain which is burgundy, brown and black with the ratio tested.

3.1.3 Selection of Ratio

The selection of ratio was done based on the high intensity of the colour given on the cotton. The

summarized table with different ratios and the intensity of the colour is shown below in Table 6 by classifying it as good, less good, and not good for burgundy, brown and black.

Table 6. Intensity of the colour

Material	Colour	Ratio	Intensity
Cotton	Burgundy	1:1:1	+
		1:2:1	++
		2:1:1	+++
	Brown	1:1:1	+
		2:1:1	+++
		1:2:1	++
	Black	1:1:1:1	++
		1:2:1:1	+
		1:1:2:1	+
		2:1:1:1	++
1:2:2:1		++	
2:2:1:1		+++	
2:1:2:1		+	

+Not good; ++ Less good; +++ Good

The combination of cinnamon, hibiscus, and fenugreek for burgundy colour, in a 2:1:1 ratio, was chosen because it gave a high intensity of colour. For the brown colour, the combination of herbs used was cinnamon, henna, and fenugreek with a ratio of 2:1:1. And for the black colour the combination of herbs was noni, eclipta, hibiscus and fenugreek and the ratio of 2:2:1:1 was selected with high intensity. The summary of the ratio is mentioned in Table 7.

Table 7. Ratio staining of the components

Composition	Colours
Cinnamon: Hibiscus: Fenugreek(2: 1: 1)	Burgundy
Cinnamon: Henna: Fenugreek (2: 1: 1)	Brown
Noni: Eclipta: Hibiscus: Fenugreek (2: 2: 1: 1)	Black

3.2 Formulation of Hair Dye

A few trials with different ratios were tried for burgundy, brown and black by increasing the ratio of each herb and increasing the time of penetration from 1 hour to 4 hours, respectively. Since the staining was not so good on the hair, the formulation was added with oils, beeswax and Carbopol gel separately to check good penetrability and intensity of the colour. For brown shade, the Carbopol gel was replaced with Aloe vera gel. Herbal hair dye was formulated as cream and gel. Formulations F1, F2, F3, F4, F7, and F8 were selected to be formulated as a cream base and formulations F5 and F6 were selected to be in a gel base. Table 8 shows the ratio of finalized herbal hair dye formulation.

Table 8. Ratio of finalized hair dye formulation

Sample Number	Ratio	Formulation
F1	CE: H: HS: F: BEESWAX 2: 6: 6: 1: 2	Cream
F2	N: E: HS: F: CO 10: 4: 2: 1: 2	Cream
F3	HS: CS: F: CO 12: 6: 1: 2	Cream
F4	CE: H: HS: F: B.WAX 2: 6: 4: 1: 2	Cream
F5	CE: H: HS: F: B.WAX: CARBO 2: 6: 6: 1: 2: 6	Gel
F6	HS: CS: F: B.WAX: CARBO 12: 6: 1: 2: 6	Gel
F7	HS: CS: F: AO 12: 3: 1: 2	Cream
F8	HS: CS: F: B.WAX 12: 6: 1: 2	Cream





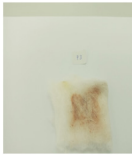

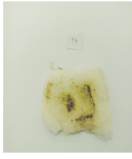







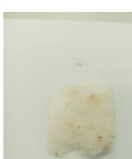
CE: Cinnamon Ethanol; H: Henna; HS: Hibiscus Specific; N: Noni; E: Ecliptaalba; CO: Coconut Oil; CS: Cinnamon Specific; F: Fenugreek; AO: Almond Oil; CARBO: Carbopol 934; B.wax: Beeswax

3.3 In Vitro and In Situ Studies for Finalized Hair Dye Formulation

In vitro and *in situ* testing was done for the finalized eight herbal hair dye formulations. *In vitro*, testing was done on the cotton while *in situ* testing was done on human

white hair. The stains obtained on the cotton and hair samples were almost the same. the results of *in vitro* and *in situ* studies of formulated hair dye are tabulated in Table 9.

Table 9. *In vitro* studies of formulated hair dye after wash

Sample No	Cotton	Hair Sample	Shade of Hair Dye
F1			Light Blonde
F2			Pure Blonde
F3			Rose Gold
F4			French Olive
F5			Swedish Blonde
F6			Pearl Platinum
F7			Smoky Pink
F8			Champagne Blonde

3.4 Accelerated Stability Studies

Accelerated stability studies were conducted to determine the physical stability and preliminary microbial detection of the eight finalized herbal hair dyes and the results are tabulated in Table 10.

Table 10. Accelerated stability studies

Sample Numbers	Physical Evaluation		Preliminary Microbial Detection	
F1	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F2	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F3	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F4	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F5	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F6	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F7	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		
F8	Colour	X	Discoloration	--
	Odor	X		
	Texture	X	Texture alteration	--
	Smoothness	X		

X: No change in Physical evaluation; --absence of microbial contamination

4. Discussion

4.1 Formulation of Hair Dye

Individual tests were done on cotton to check the staining of each plant for a particular colour. Hence, the selected herbs extractions which gave more colorant on the cotton, were further tested for combination tests with other herbs. When hibiscus was extracted with methanol, the colour obtained was red. At the same time, when extracted cinnamon was mixed with methanol, the colour obtained was like burgundy. Therefore, the combination of both hibiscus and cinnamon extracted with methanol was selected to form a burgundy colour as a new formulation for the hair dye.

All the plants give good colours and stains properly on cotton and also on the hair sample. However, the green colour that was supposed to have been from pandan leaves that were extracted with water, ethanol, and methanol was not satisfactory for the individual tests. Therefore, the formulation for green colour hair dye had to be dropped and was not used for further testing.

Based on the finalized ratio, multiple trials with different ratios were conducted. Numerous trials were conducted from a fixed ratio of all the colours by increasing the ratio of each herb and by increasing the penetration time from 1 hour to 4 hours. The formula was modified based on a trial-and-error base for all the primary colours. The formulation was tested with various ratios until a better colour was formed. However, the formulation did not stain the hair after washing with tap water without the aid of shampoo. Hence, the formulation was improved with the addition of almond oil, coconut oil, beeswax and carbopol gel for better penetrability, and this formulation was later improved to a cream and gel base.

4.2 In Vitro Studies

The hair dye was first tested using cotton wool because it has the same properties as hair. For black shade hair dye, the plants used were noni, *Eclipta alba* L., hibiscus, and fenugreek for cleansing purposes. Unfortunately, the colour did not stain properly even on cotton or on hair samples even though the ratio was manipulated to the maximum. The formulation for black showed a greyish colour in the hair but not on the cotton.

The finalized formulation of F5 and F6 shows that gel formulation gave good staining and the colour

efficiency was better compared to other ratios, which were tried with different oils. Meanwhile, F1, F2, F3, F4, F7 and F8 showed that cream formulation gave good staining and better colour intensity with the addition of oils in the cream base. Cotton and hair samples got better colour based on the staining. The finalized naming was discovered through pinterest.com by mixing and matching the hair sample for the naming of the last eight different shades. The eight formulations were given names starting from F1 as Light Blonde, F2 as Pure Blonde, F3 as Rose Gold, F4 as French Olive, F5 as Swedish Blonde, F6 as Pearl Platinum, F7 as Smoky Pink and F8 as Champagne Blonde.

4.3 Accelerated Stability Studies

Based on the results of the sample product for accelerated studies, showed that the eight formulated hair dyes did not have any microbial contamination or fungus growth. The product remained the same as how it was kept inside the humidity chamber and there were no alterations in the texture. Moreover, for the physical evaluation, the colour remained the same without changes for all eight formulations. There was no bad or pungent odour when the cap of the jar was opened, and the formulation stayed in good texture. The formulation was smooth enough and consistency of a gel and cream.

5. Conclusion

As a conclusion of the study, it was found out that eight different shades of the semi-solid formulation were obtained for the formulation of natural herbal hair dye. All those extracted herbs and plants were tested for physical and colour intensity in cotton as well as in the hair. Almost all the tests passed according to the hypothesis where all the hair dye was stained in the hair. Currently, some of the products available in the market may not be suitable for some religious reasons. Some of the hair dyes are not halal-certified due to the ingredients used. Using herbal ingredients guarantees a halal process of making it as it will only be extracted using alcohol and is completely evaporated and no other chemicals that could raise the suspicion of the user were added. In addition, it is suitable for natural hair colourants for all age groups. Accelerated stability studies in terms of colour intensity are

recommended for further studies. These formulations are recommended for further studies to be formulated as a hair dye as shampoo together with a conditioner, a spray hair dye, or a foam type of hair dye.

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