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Research Article

Formulation and Evaluation of Herbal Nanoemulgel Hair Dye Incorporating Hibiscus and Henna Extracts

Pavithra T.^{*}, Manoj M., Namratha G. M., Priyanka M. S., Ranjitha B. K., Yashaswini H.

Bharathi College of Pharmacy, Bharathinagara, Mandya – 571422, Karnataka, India.

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ABSTRACT

The present study focuses on the formulation and evaluation of a herbal nanoemulgel hair dye using *Hibiscus rosa-sinensis* and *Lawsonia inermis* (henna) extracts as natural colouring agents. Conventional hair dyes may contain synthetic chemicals that can cause irritation or damage to hair and scalp. Therefore, herbal formulations are gaining attention as potential alternatives for natural hair colouring. Nanoemulgel combines the advantages of a nanoemulsion and a gel system, offering improved consistency, spreadability, and uniform application. In this study, aqueous extracts of hibiscus and henna were incorporated into a formulation containing coconut oil, Tween 20, acacia, glycerin, and hydroxypropyl methylcellulose (HPMC). The prepared formulations were evaluated for organoleptic properties, pH, physical stability, spreadability, sensitivity, and excipient compatibility. The formulations exhibited a reddish-pink colour, characteristic odour, good consistency, and homogeneity. The measured pH values ranged from 7.0 to 7.4, and no visible phase separation was reported during the observed storage period. The formulations also showed good spreadability, while no visible sensitivity reactions were reported under the preliminary evaluation conditions. The findings suggest that hibiscus–henna herbal nanoemulgel may have potential for application as a natural hair-colouring formulation. Further studies, including droplet-size analysis, comprehensive stability testing, and detailed safety evaluation, are required to establish its quality, performance, and suitability for practical use.

***Corresponding Author:** Pavithra T

Address: Bharathi College of Pharmacy, Bharathinagara, Mandya – 571422, Karnataka, India

Email ✉: pavipraphu@gmail.com

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INTRODUCTION

Introduction of Nano-emulgel:

Nano-emulsions are heterogeneous colloidal mixtures of oil and water, with one component as a dispersed phase and the other as a continuous phase. A surfactant known as an emulsifier is adsorbed at the interface between the dispersed and continuous phases, lowering the surface tension and thus stabilizing the system. These systems possess high thermodynamic stability leading to longer shelf life compared to simple emulsions, micelles or suspensions, etc. Despite having various advantages, nano-emulsions are limited by their low viscosity leading to low retention time and spread ability.¹

These problems can be resolved by modifying nano-emulsion into a nano-emulgel by using a suitable gelling agent.²

Emulgels for dermatological use have several favorable properties such as being thixotropic, greaseless, easily spreadable, easily removable, emollient, long shelf-life surfactant³ Natural dyes have been used since ancient times, when they were used not only for hair coloration, but also for medicinal, decoration and religious purposes.^{4,5,6} In the early days, hair dyes were obtained from metallic compounds, plant extracts, dried plants or their mixtures.⁷ Before the invention of first synthetic aniline dye, mauve, in 1856, different plant extracts and herbal preparations such as mullein, birch bark, turmeric, and saffron have been used for hair dyeing.

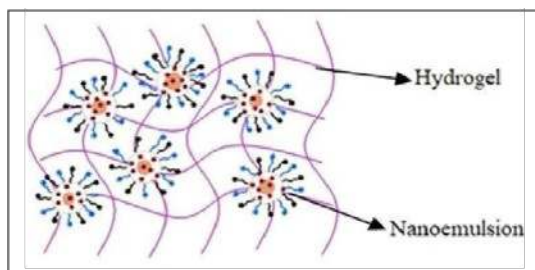


Figure 1.1: Structure of Nanoemulgel.⁸

Nano emulgel Hair dye:

Nanoemulgel hair dye is an advanced topical hair-coloring formulation that combines a nanoemulsion (sub-micron oil droplets dispersed in water) with a gel matrix. It improves dye penetration into the hair shaft and

cuticle via high surface area and tiny droplet size, offering superior color retention, non-dripping consistency, and reduced chemical damage. Key Characteristics & Composition Oil Phase: Contains penetration enhancers, essential or mineral oils, and hydrophobic dye intermediates or natural pigments. Aqueous Phase: Water mixed with water-soluble components, buffers, and anti-oxidants. Surfactants/Cosurfactants: Stabilize the nanodroplets (typically droplet sizes between 20 nm and 500 nm) and lower interfacial tension. Gelling Agent: Polymers like Carbopol or HPMC that convert the liquid nanoemulsion into a spreadable, non-messy gel. Advantages Over Traditional Dyes Enhanced Deposition: Nano-sized droplets fit better into microscopic gaps of the hair cuticle, ensuring deeper and more uniform pigment uptake. Better Stability: The dual system prevents phase separation, creaming, and sedimentation that plague regular liquid emulsions. Improved Spreadability: Spreads smoothly along individual hair strands without running or dripping onto the scalp. Reduced Irritation: Controlled release and localized action minimize direct aggressive contact with the scalp skin.⁹

INGREDIENTS PROFILE

1. HIBISCUS:

Synonym: China rose, shoe flower, Chinese hibiscus

Biological Source: consists of the fresh or dried flowers of *Hibiscus rosa-sinensis* L.

Botanical name: *Hibiscus rosa-*

Family: Malvaceae.



Figure 1.2: Picture of Hibiscus.



Chemical Constituents:

Hibiscus contains various phytochemicals including: Flavonoids (Quercetin, Apigenin), anthocyanins (Pelargonidin), tannins, saponins, terpenoids, glycosides, alkaloids, vitamins (vitamin c, riboflavin, thiamine).

Uses:

- Natural hair dye and hair growth promoter.
- Used in hair oils, shampoos, and herbal hair formulations.
- Use traditionally for wound healing and skin care.
- Helps reduce dandruff and strengthens hair roots.

Possesses antioxidant and antimicrobial properties.

1. HENNA:

Synonym: Mehndi, Henna, *Lawsonia alba*.

Biological Source: Henna consists of the dried leaves of *Lawsonia inermis* L., a shrub

or small tree belonging to the family Lythraceae

Botanical name: *Lawsonia inermis* L.

Family: Lythraceae.



Figure 1.3: Picture of Henna.

Chemical Constituents:

Lawsonic acid (2-hydroxy-1,4-naphthoquinone – principal coloring matter), flavonoids, tannins, phenolic glycosides, coumarins, xanthenes, saponins, alkaloids, terpenoids, β -sitosterol, and stigmasterol.

Uses: Natural hair dye, body art (mehndi), coloring nails, dyeing fabrics and leather, antimicrobial activity, antifungal activity, wound healing, anti-inflammatory action, treatment of skin disorders, cooling agent, and cosmetic applications.

MATERIALS AND METHODOLOGY

EXTRACTION PROCEDURE:

1. Weighing of Drug:

Accurately weigh 50 g of powdered hibiscus and henna flowers and use solvent as distilled water (Q.S).

1. Decoction:

- Take the dried and coarsely powdered crude drug.
- Add a specified quantity of water (commonly 1 part drug with 10–20 parts water).
- Boil the mixture gently for a specified period, usually 15–30 minutes.
- Continue heating until the volume is reduced to the required amount.
- Allow the mixture to cool.
- Filter the extract using muslin cloth or filter paper.
- Collect the filtrate (decoction) and use it immediately or concentrate it further if required.

2. Filtration:

- Allow the decoction to cool to room temperature.
- First filter through a clean muslin cloth to remove large plant particles.
- Then filter the extract using Whatman filter paper No. 1 for clearer filtrate.
- Collect the clear filtrate in a clean, dry container.
- If the extract is very thick, dilute slightly with distilled water before filtration.
- Decoction → Cooling → Muslin cloth filtration → Whatman No. 1 filter paper → Clear extract.

METHOD OF PREPARATION:

Step 1 — Prepare the herbal extracts

- Henna extract
- Henna powder: 10 g
- Purified water: 100ml
- Macerate with occasional stirring for 2–4 hours.
- Warm gently at about 50–60°C for 30 minutes.
- Cool and filter thoroughly.



- Hibiscus extract
- Hibiscus powder/petals: 10 g
- Purified water: 100 mL
- Macerate for 2–4 hours.
- Warm gently at about 50–60°C for 30 minutes.
- Cool and filter.
- For the formulation, use 5 g of henna extract and 2.5 g of hibiscus extract.

Step 2 — Prepare the acacia solution

- Take approximately 8–10 mL purified water.
- Slowly add 1 g acacia with continuous stirring.
- Allow it to hydrate for approximately 30–60 minutes.
- Stir until a smooth dispersion is obtained.
- If there are large particles, filter the dispersion before use.
- Do not put the acacia directly into coconut oil.

Step 3 — Prepare the aqueous herbal phase

- In a beaker, combine:
- Henna extract — 5 g
- Hibiscus extract — 2.5 g
- Glycerine — 2.5 g
- Prepared acacia dispersion — containing 1 g acacia
- Mix gently until uniform.
- Add part of the remaining purified water as required.

Step 4 — Prepare the oil phase

- In a separate beaker:
- Coconut oil — 2 g
- Tween 20 — 2 g

- Mix thoroughly.
- If the coconut oil is not mixing properly with Tween 20, warm gently in a water bath at approximately 35–40°C while stirring.
- You should obtain a uniform oil/surfactant mixture without visible lumps

Step 5 — Prepare the primary emulsion

This is the important step because your previous batch was separating. Place the aqueous herbal phase under the homogenizer. Start homogenization at a moderate speed. Add the oil phase very slowly to the aqueous phase. Continue homogenization during the addition. Once all the oil phase has been added, homogenize for approximately 5 minutes. Avoid creating a strong vortex because it introduces air and produces excessive foam. The mixture should become a uniform creamy/brownish emulsion.

Step 6 — Prepare HPMC gel separately For 1.5 g HPMC:

Take approximately 10–15 mL purified water. Sprinkle HPMC slowly into the water while stirring. Continue stirring to disperse it uniformly. Allow it to hydrate for 30–60 minutes, or according to the specification of your particular HPMC grade. Continue gentle mixing until a smooth, lump-free gel is

obtained. Do not add triethanolamine to HPMC just to make the gel. HPMC does not require neutralization like Carbopol.

Step 7— Convert nanoemulsion into nanoemulgel.

Take the hydrated HPMC gel. Add the prepared herbal nanoemulsion slowly in portions. Mix gently after each addition. Continue until the mixture becomes homogeneous. Avoid high-speed homogenization at this stage because it can introduce excessive air and disturb the gel structure.¹²



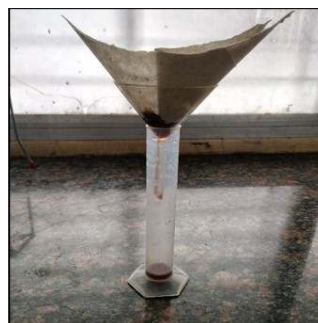
DRIED FLOWER OF HIBISCUS.



DRIED LEAVES OF HENNA.



BOILING



FILTRATION



ADDING GLYCERINE



HOMOGENIZING



TESTING OF PH



FINAL PRODUCT

PROCEDURE FOR EVALUATION TEST

FIGURE 1.4: PROCEDURE OF NANO EMULGEL

1. Physical Appearance

Purpose: To evaluate color, Odor, homogeneity, consistency, and phase separation.

Procedure:

- Visually inspect the formulation for color, Odor, smoothness, and homogeneity.
- Check for any phase separation or grittiness.

2. pH Determination

Purpose: To ensure scalp compatibility.

Procedure:

- Dissolve 1 g of nanoemulgel in 10 mL distilled water.
- Measure pH using a calibrated digital pH meter.
- Take three readings and calculate the average.

3. Spreadability

Purpose: To evaluate ease of application on hair and scalp.

Procedure:

- Place 1 g of gel between two glass slides.
- Apply a 100 g weight for 5 minutes.
- Measure the time required for the upper slide to move a fixed distance.

Formula:

$$S = \frac{M \times L}{T}$$

Where:

S = Spreadability M = Weight (g)

L = Length moved (cm) T = Time (sec)

4. Dyeing Efficiency Test

Purpose: To evaluate coloring ability.

Procedure:

- Apply nanoemulgel on grey hair strands.
- Leave for 30–60 minutes.
- Wash and dry the hair strands.
- Observe color intensity and uniformity.

5. Washability Test

Purpose: To evaluate ease of washing from hair.

Procedure:

- Apply formulation on hair strands.
- Wash with water after drying.
- Observe ease of removal.

6. Skin Irritation Test

Purpose: To assess safety for scalp use.

Procedure:

- Apply a small amount on the forearm.
- Observe for redness, itching, or irritation after 24 hours.¹³

Table 1: Biological Profile of the Selected Plants^{10,11}

SL NO.	Plant / Ingredients	Biological name	Family	Major constituents	Role in formulation
1	Henna	<i>Lawsonia inermis L</i>	Lythraceae	Lawsonic acid, tannins, flavonoids	Primary natural Hair colouring agent
2	Hibiscus	<i>Hibiscus rosa-Sinensis L</i>	Malvaceae	Anthocyanins, flavonoids, Mucilage, tannins	Provide phytochemicals And may contribute to formulation



SL NO.	Plant / Ingredients	Biological name	Family	Major constituents	Role in formulation
3	Tea	<i>Camellia sinensis(L) kuntze</i>	Theaceae	Catechins, tannins, Polyphenols, caffeine	Provides polyphenols And may contribute brownish tones
4	Coconut oil	<i>Cocos nucifera L</i>	Arecaceae	Lauric acid, myristic acid and other fatty acids	Oil phase of the nanoemulsion
5	Tween 20	Polysorbate 20	Non-ionic surfactant	Polyoxyethylene sorbitan monolaurate	Emulsifier; helps stabilize Oil droplets
6	Glycerin	Glycerol	Humectant	Glycerol	Retains moisture and improves spreadability
7	Carbopol 940	Cross-linked Polyacrylic acid polymer	Synthetic gelling agent	Cross-linked polyacrylate	Forms moisture And increases viscosity
8	Triethanolamine	<i>TEA</i>	Ph-adjusting agent	Triethanolamine	Neutralises Carbopol and Helps from the gel
9	HPMC	Hydroxypropyl methylcellulose	Cellulose-Derived polymer	Modified cellulose polymer	Alternative gelling and viscosity-enhancing agent
10	Acacia	<i>Senegalia Senegal (L) Britton</i>	Fabaceae	Arabinogalactan-rich polysaccharides	Natural thickener and stabiliser
11	Distilled water	Purified water, H ₂ O	Aqueous vehicle	Water	Solvent and continuous aqueous phase

RESULTS AND DISCUSSION

1. EVALUATION PARAMETER

The prepared herbal nano emulgel hair dye was evaluated for various parameters such as organoleptic

properties, pH, phase separation, dyeing effect test, Irritancy and Compatibility Test, spread ability.

1.1 ORGANOLEPTIC PROPERTIES

The Organoleptic properties of the formulated herbal nano emulgel hair dye were judged by colour, odour, consistency and Homogeneity.

Table 2: Result of Various Evaluation Parameters

Sl no	Parameters	Formulation 1	Formulation 2	Formulation 3
1.	Organoleptic properties			
	Colour	Reddish pink	Reddish pink	Reddish pink
	Odour	Characteristic	Characteristic	Characteristic
	Consistency	Good	Good	Good
	Homogeneity	Good	Good	Good

Test for pH

Good pH test is performed by using pH paper range in between 7-7.3 hence the nano emulgel herbal dye should be slightly alkaline range



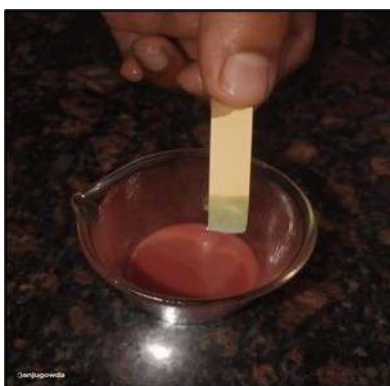
Figure 5.1 picture of p^H

Table 3: pH of Formulation F1 to F3 Formulation

Sl no	Formulation	pH
1.	F1	7
2.	F2	7.1
3.	F3	7.4

Table 4: Formulation for Herbal Nano Emulgel Hair Dye

Sl no	Ingredients	F1	F2	F3	Functions
1	Henna extract	4g	4.5g	5g	Primary natural coloring agent. Contains lawsone, which binds with hair keratin and imparts a reddish-brown color. It also conditions hair, reduces dandruff, and strengthens hair fibers.
2	Hibiscus extract	2g	2g	2.5g	Acts as a natural hair conditioner and antioxidant. Helps improve hair growth, reduces premature graying, adds shine, and enhances the overall coloring effect.
3	Glycerin	2.5g	2.5g	2.5g	Humectant and moisturizer. Retains moisture in the formulation and prevents hair dryness after dye application. Improves smoothness and spreadability of the nanoemulgel
4	Acacia	1g	1g	1g	Natural emulsifier and stabilizer. Helps stabilize the emulsion system and increases viscosity, improving consistency and adhesion of the dye to hair.
5	Coconut oil	2g	2g	2g	Oil phase component. Nourishes and conditions hair, reduces protein loss, improves softness, and facilitates penetration of active constituents into hair shafts.
6	Tween 20	2g	2g	2g	Non-ionic surfactant/emulsifying agent. Reduces interfacial tension between oil and water phases, helping to form and stabilize nano-sized emulsion droplets.

Sl no	Ingredients	F1	F2	F3	Functions
7	HPMC	0.5g	1g	1.5g	Gelling agent. Converts the nanoemulsion into a nanoemulgel, provides suitable viscosity, improves spreadability, and enhances residence time on hair.
8	Purified water	33.5g	33.5g	33.5g	Vehicle and solvent. Dissolves hydrophilic ingredients and forms the aqueous phase of the nanoemulsion.
9	TEA	q. s for pH	q. s for pH	q. s for pH	Rich in tannins that act as a natural color enhancer. It deepens and darkens the hair color and improves dye retention on hair.

PHASE SEPARATION

The herbal nano emulgel hair dye was stored at room temperature and observed for physical stability. No phase separation, precipitation, or visible changes were noted during the study period, indicating that the formulation remained stable at room temperature

SENSITIVITY AND IRRITANCY TEST

The formulated herbal nano emulgel hair dye doesn't show any type of sensitivity reaction. The formulated herbal nano emulgel hair dye shows no redness, edema, irritation, or inflammation during studies. The formulated herbal nano emulgel hair dye.



Figure 5.2 picture of sensitivity and irritancy test

Table 4: Sensitivity and Irritancy Test

Sl no	Formulation	Sensitivity	Erythema	Edema	Irritation	Inflamtion
1.	F1	NO	NO	NO	NO	NO
2.	F2	NO	NO	NO	NO	NO
3.	F3	NO	NO	NO	NO	NO

DRUG- EXCIPIENTS COMPATABILITY TEST

Stability, Appearance, Safety, Efficacy.

Purpose of Drug-Excipients Compatibility Testing: To ensure no interaction occurs between hibiscus, henna and the excipients that may affect:

Table 5: Drug- Excipients Compatibility of Formulation F1 to F3

Sl no	Excipients	Compatibility with drug hibiscus and henna
1.	Henna extract	Good
2.	Hibiscus extract	Very good
3.	Glycerin	Good



Sl no	Excipients	Compatibility with drug hibiscus and henna
4.	Acacia	Very good
5.	Coconut oil	Very good
6.	Tween 20	Very good
7.	HPMC	Good
8.	Purified water	Very good
9.	Tea	Good

SPREADABILITY:

The hibiscus and henna of herbal nano emulgel hair dye showed good spreadability when applied to the skin. It spread smoothly and evenly with gentle rubbing, forming a thin and uniform layer. The hair dye was easily absorbed without leaving any greasy or sticky feeling. It provided a soft and smooth texture on the skin surface, indicating good consistency and ease of application. Overall, the nano emulgel exhibited excellent spreadability suitable for topical use.



Figure 5.3: Picture of Spreadability

The spreadability of nano emulgel herbal hair dye was found to be 100g.cm/s.

DYEING EFFICACY TEST:

The prepared herbal nanoemulgel hair dye exhibited good dyeing efficacy, imparting uniform colour and improved colour intensity to the hair strands.

WASHABILITY TEST:

The prepared herbal nanoemulgel hair dye showed good washability with satisfactory colour retention after washing.

CONCLUSION:

The herbal nanoemulgel hair dye incorporating henna (*Lawsonia inermis*) and hibiscus (*Hibiscus rosa-sinensis*) was successfully formulated and evaluated. The prepared formulations showed a reddish-pink colour, good consistency, homogeneity, and satisfactory spreadability. The pH values ranged from 7.0 to 7.4, and no visible phase separation was observed during the study period. Preliminary sensitivity testing showed no visible signs of irritation under the test conditions. The formulation shows potential as a natural alternative to conventional hair dyes. However, further studies on droplet size, long-term stability, dyeing efficiency, and safety are required to confirm its suitability for practical use.

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