FORMULATION AND EVALUATION OF LIPSTICK USING IXORA COCCINEA FLOWER EXTRACT AS A NATURAL COLORING AGENT

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ABSTRACT

Objective: The objective of the present research work was to formulate and evaluate lipstick using fresh flowers of *Ixora coccinea* extract as a coloring agent.

Methods: Fresh flowers of *I. coccinea* were collected, and coloring pigment was extracted by cold maceration technique using ethanol as solvent. Lipstick formulations (F1-F4) were prepared using beeswax, cocoa butter, carnauba wax, white petroleum jelly, coconut oil, and other excipients. The prepared formulations were evaluated for various evaluation parameters such as color, texture, pH, melting point, breaking point, hardness, spreadability, and aging stability.

Results: The results of different evaluation parameters of prepared lipstick formulations were within the standard range. Formulation (F3) was good enough to meet the general characteristics for ideal lipstick as all the evaluation parameters met the standard values of lipsticks.

Conclusion: The natural dye obtained from the petals of the *I. coccinea* flower plant was successfully used as a coloring agent in the formulation of lipstick in this study. Results of the study confirmed that this color pigment obtained from fresh flowers of *I. coccinea* may prove to be a useful choice in the formulation of lipstick. Natural coloring agents are safer options and are a better choice compared to their synthetic counterparts in cosmetic formulations.

Keywords: Herbal cosmetics, *Ixora coccinea*, Lipstick, Natural pigments.

INTRODUCTION

Cosmetics are made from mixtures of chemical compounds derived from either natural or synthetic sources. Cosmetic items have mild action on the human body for the purpose of cleaning, beautifying, adding attractiveness, altering appearance, or keeping or promoting the skin or hair in good condition. Cosmetics are classified according to their application on the skin such as creams, powder, nylons such as nail polish, manicure preparation, oral cavity dentifrices, mouth-washes, eye cream, eye-lashes for eyes, and shampoo for hair. Based on their function, they are classified as curative actions such as antiperspirants, protective function face powder, and decorative function lipstick, nail polish [1,2].

The name herbal is a symbol of safety compared to the synthetic one which has side effects on human health. Herbal cosmetics have gained a demand in the world market and are a helpful skill of nature. There is an extensive variety of herbal cosmetics products to satisfy the beauty of an individual. Including herbal ingredients in cosmetics is much safer for the skin compared to synthetic ingredients. Herbal lipsticks are mainly formulated to overcome the disadvantages of synthetic lipsticks owing to their minimal side effects. Herbal Lipsticks have several advantages over their synthetic counterparts. Fewer side effects, more choices for color, texture, and luster make them more reliable [2,6]. Thus, the use of herbal colorants in cosmetic preparations will surely help to overcome the side effects associated with synthetic colorants. Synthetic cosmetics mainly contain chemicals and have many side effects such as drying of lips, discoloration of lips when used for a long time, and reported to cause lip cancer [1,3]. Lipsticks are small solid stick formulated to impart color and soothing effect to the lip. Lipsticks are used for many purposes such as coloring lips, for providing protection, and softening the lips. *Ixora coccinea* is a flowering plant of the family Rubiaceae. It is also known as Raktaka, rukmine [4]. It has medicinal importance in a traditional system. Raktaka is used in curing varies ailments such as dysentery leukorrhea activity and anti-inflammatory [5,6]. *I. coccinea* has therapeutic potential due to presence of bioactive compounds such as flavones, isoflavones, flavonoids, anthocyanins, coumarins, lignans, catechins, isocatechins, alkaloids, tannin, saponins, and triterpenoid [5]. It has been reported to contain several color pigments such as anthocyanins, betalains, cyanidin, carotenoids, xanthophyll, phycocerythrin, and phycocyanin [5].

In the present research work, an attempt is made to extract coloring pigment from the *I. coccinea* plant and use it as a natural coloring agent in the formulation of lipsticks.

METHODS

The materials used were either analytical research grade or lab research grade. *I. coccinea* flowers were collected from the medicinal garden of Rani Chennamma College of Pharmacy, Belagavi, Karnataka. Ethanol was purchased from nice chemicals Pvt. Ltd., Kochi, Beeswax from Veda oils, Carnauba wax, and Cocoa butter from Burgoyne Burblings and Co. Mumbai.

Plant collection and authentication

The flowers of the plant *I. coccinea* (Family: Rubiaceae) were collected from the medicinal garden of Rani Chennamma College of Pharmacy, Belagavi, Karnataka [Fig. 1] and authenticated (Assesement No: RMRC-1651) by Dr. Harsha Hegde, Taxonomist, ICMR Belagavi, Karnataka. India.

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Preparation of ethanol extract
About 20 g of fresh flowers of *I. coccinea* were collected and washed 3 times with double distilled water to remove dust particles. The flowers were transferred to the beaker containing 100 ml of ethanol and kept for maceration for 24 h followed by filtration with muslin cloth followed by Whatman filter paper no.1. The filtrate was collected in a china dish and placed in water bath [7].

Phytochemical test of *I. coccinea* extract
Qualitative chemical tests were conducted for the extract to identify the various phytoconstituents [7,8].

Tests for carbohydrates
*Molisch’s test*
To the extract, a few drops of alpha-naphthol solution in alcohol were shaken and 1–2 ml of concentrated sulfuric acid was added from the sides of the test tube. A violet ring was observed at the junction of two liquids.

*Fehling’s test*
About 1 ml each Fehling’s A, Fehling’s B solution was mixed, the mixture was boiled for 1 min, and an equal volume of test solution was added. The mixture was heated in a boiling water bath for 5–10 min and observed for a yellow and then brick-red precipitate.

Tests for proteins
*Biuret test (General test)*
About 3 ml of extract 4% sodium hydroxide was added and a few drop of 1% CuSO4 solution and observed for violet or pink color.

*Million’s test (for proteins)*
About 3 ml of extract and 5 ml Million’s reagent was added, to get white precipitate and warm, the precipitate turns to brick red or the precipitate dissolves giving red colored solution.

Tests for steroid
*Salkowski reaction*
About 2 ml of the extract was added to 2 ml chloroform and 2 ml concentrated H2SO4. Chloroform layer appeared red and acid the layer showed greenish-yellow fluorescence.

Tests for glycosides
*Test for deoxysugars (Keller Killani test)*
About 2 ml extract, add glacial acetic acid, one drop of 5% FeCl3, and concentrated H2SO4 on the side of a test tube. Reddish-brown color was observed at the junction of the two liquids and bluish-green colors to the upper layer.

*Legal’s test*
To the test solution, add 1ml of pyridine and 1ml of sodium nitroprusside and observe for pink to red color.

*Foam test*
The drug extract or dry powder was shaken vigorously with water. The persistent foam was observed.

Tests for flavonoids
*Shinoda test*
To the extract, add 5 ml of 95% ethanol, a few drops of concentrated HCl, and 0.5 g magnesium. Orange, pink, red to purple color has appeared.

*Fig. 1: Flowers of the plant Ixora coccinea*

*Fig. 2: Schematic presentation of lipstick preparation*
Tests for alkaloids

**Wagner’s test**

About 2 ml of the extract was added to a few drops of Wagner’s reagent and observed for reddish-brown precipitate.
**Tannic acid test**
The test solution is treated with a tannic acid solution which gave buff-colored precipitate.

**Tests for tannins and phenolic compounds**
3 ml of extract was added to a few drops of the following reagents;
- a. Gelatin solution: White precipitate was observed.
- b. Lead acetate solution: Gave white precipitate.
- c. 5% FeCl3 solution: Deep blue-black colored was observed.
- d. Bromine water: There was decoloration of bromine water.
- e. Acetic acid solution: Red color solution was observed.

**Formulation of lipstick**
The first phase of the formulation was prepared by melting beeswax, cocoa butter, carnauba wax, white petroleum jelly in a China dish on an electrical water bath with decreasing order of their melting point. The second phase was prepared by mild heating of coconut oil and extract both the phases are mixed together at the same temperature and vanilla essence was added. Then, the mixture was poured into the lipstick mold lubricated with the liquid paraffin mold was kept on an ice bath for solidification and after 2–3 min an excess amount of mixture was scraped off using a metal scraper. Lipsticks were demolded after 5–6 min four formulations were prepared, F1 to F4 as shown in Table 1 [1,3,9] Schematic presentation of lipstick preparation is given in Fig 2. Formulated lipstick formulation from F1-F4 are shown in Fig. 3.

**Evaluation**

**Color and texture**
Formulated lipsticks were checked visually for color, glossy and smooth texture [6].

**Melting point**
Determination of melting point is important as it is an indication of the limit of safe storage [10]. The melting point of formulated lipstick was determined by the capillary tube method. The capillary was filled, kept in the capillary apparatus and observed the product for its melting. The procedure was performed for 3 times and the melting point ratio was observed for the prepared formulations [11,12].

**Breaking point**
Breaking point was done to determine the strength of lipstick. The lipstick was held horizontally in a socket inch away from the edge of support. The weight was gradually increased by a specific value (10 g) at specific interval of 30 s and weight at which the sample breaks was considered as the breaking point [6,10] [Fig. 4].

**Surface anomalies**
Any defects on the surface were observed, such as the formation of crystals contamination by mold [6].

**Determination of hardness**
Formulated lipstick from each formulation was selected randomly and hardness was measured using Monsanto hardness tester. The average result of each formulation was calculated and noted [10] [Fig. 5].

**Determination of spreadability**
The test was done by repeatedly applying the formulation onto the glass slide to observe the uniformity in the formulation whether it sticks, leave fragments, deformed or, is broken during application [10].

**Aging stability**
All the formulations were placed in a china dish and kept at 40° C in a hot air oven for 1 h and various parameters such as bleeding, crystals onto the surface and ease of application were checked [6].

**Solubility test**
1 g of the sample was taken in different test tubes containing the different solvents. After the addition of each portion of solvent, test tubes were shaken vigorously and then observed visually [6].

**pH**
The pH of formulated herbal lipsticks was determined using pH meter [6,12].

**Results**

**Total yield**
20 g of fresh flowers of I. coccinea in 100 ml of ethanol gave 2 ml of a concentrated extract [Fig. 6].

**Phytochemical test of I. coccinea extract**
The extract of I. coccinea was subjected for qualitative analysis: The extract showed the presence of carbohydrates, flavonoids, alkaloids, tannins, phenols, steroids and proteins were present. The results are depicted in Table 2.

The results of qualitative screening of phytochemicals of I. coccinea flower extract revealed the presence of diverse types of phytochemicals namely, alkaloids, tannins, phenolic compounds, carbohydrates, proteins, glycosides and flavonoids [4].

**Color and appearance**
The prepared herbal lipstick containing I. coccinea flower extract used as a natural pigment was inspected visually for their color and appearance which was found to be a magenta color with glossy texture [Fig. 7].

**Melting point**
The F1 to F4 formulation showed a melting point in the range of 61° C to 65° C.

**Table 1: Formulation of lipstick (F1-F4)**

| Ingredients                        | Formulation |
|------------------------------------|-------------|
|                                    | F1          | F2          | F3          | F4          |
| Beeswax                            | 2 g         | 2 g         | 2 g         | 2 g         |
| Cocoa butter                        | 1 g         | 1 g         | 1 g         | 1 g         |
| Carnauba wax                        | 1 g         | 1 g         | 1 g         | 1 g         |
| Yellow petroleum jelly              | 2.5 ml      | 2.5 ml      | 2.5 ml      | 2.5 ml      |
| Coconut oil                         | 0.5 ml      | 0.5 ml      | 0.5 ml      | 0.5 ml      |
| Colorant (I. coccinea extract)      | –           | 1 ml        | 2 ml        | 3 ml        |
| Vanillin essence                    | q.s         | q.s         | q.s         | q.s         |
| Citric acid                         | q.s         | q.s         | q.s         | q.s         |

**Table 2: Phytochemical screening (qualitative test) for Ixora coccinea flower**

| Name of the test                  | Ethanolic extract |
|-----------------------------------|-------------------|
| Test for carbohydrates            |                   |
| Molisch test                      | +                 |
| Fehling’s test (reducing sugar)   | +                 |
| Test for proteins                 |                   |
| Biuret test                       | +                 |
| Million’s test                    | +                 |
| Test for steroids                 |                   |
| Salkowski reaction                | -                 |
| Test for glycosides               |                   |
| Killer-Killani (cardiac glycosides)| +                 |
| Legal’s test (cardenolides)       | +                 |
| Foam test (Saponin glycoside)     | -                 |
| Test for flavonoids               |                   |
| Shinoda test                      | +                 |
| Test for alkaloids                |                   |
| Wagner’s test                     | +                 |
| Tannic acid test                  | +                 |
| Test for tannins and phenolic compounds | 5% FeCl3 Solution | + | Gelatine | + |
| Lead acetate                      | +                 |
| Bromine water                     | +                 |
| Acetic acid                       | +                 |

Note: **"** = Present; **-"** = Absent
Solubility

The solubility of the formulated herbal lipstick was found by dissolving 50 mg of lipstick in various solvents such as acetone, water, alcohol, buffer and chloroform where the results showed that all the formulations from F1 to F4 were soluble in chloroform [Fig. 8].

Surface anomalies

This is studied to determine the surface defects such as the formation of crystals on the surface, contamination by mold, fungi, and others. There were no defects found on the surfaces of the formulation.

Aging stability

Blending, streaking, catering, and blooming, parameters were checked and all the formulations of lipsticks from F1-F3 were found to be smooth after 1 h except the F4 formulation failed the aging stability test.

pH

The pH of formulation from F1 to F4 was in the range of pH 6–pH 7.

Breaking point

This test was carried out to find out the value of maximum load that lipstick can withstand before it breaks which shows the strength of the lipstick where the results revealed that all the formulations from F1 to F4 were in the range of 140–160 g.

Spreadability

It was tested by repeatedly applying the lipstick onto the glass slide to observe the uniformity in the formulation of the protective layer and whether the stick fragmented, deformed, or broke during application the results showed that all the formulations from F1 to F4 where of the intermediate result [Fig. 9].

Hardness test

Formulations from F1-F4 were selected randomly and measured using the Monsanto hardness tester. The average result of each formulation was calculated and found in the range of 1–2 kg/cm².

DISCUSSION

It is very essential to maintain a uniform standard for herbal lipstick; keeping this view in mind, the formulated herbal lipsticks were evaluated for various parameters such as color and appearance, melting point, breaking point, solubility test, pH, hardness, spreadability, aging stability and surface anomalies [Table 3]. So based on the evaluation parameters it is observed that F2 formulation has a problem of not imparting good color; this may be due to less color concentration in the formulation and F4 formulation fails the aging stability test as it causes bleeding problems due to more color concentration and even hardness is less compared to that of other formulation. Hence, among all the formulations F1–F4, F3 was good enough to meet the general characteristics for ideal lipsticks as all the evaluation parameters meet the standard values of lipsticks.

CONCLUSION

The results of the present investigation revealed that the colored pigment from the flower extract of I. coccinea can be effectively used as a natural colorant in the preparation of lipstick formulations. The use of a natural colorant will provide a suitable alternative for synthetic colorants which are known for their side effect. Thus, this colorant which has been derived from the natural origin can be effectively used in various cosmetic preparations and the side effects associated with the use of synthetic colorants can be efficiently minimized.

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Table 3: Evaluation parameters of Formulation F1, F2, F3 and F4

| Evaluation Parameter | F1 | F2 | F3 | F4 |
|----------------------|----|----|----|----|
| Color and appearance | Colorless with glossy | Light pink with slightly rough | Magenta with glossy | Magenta with slightly rough |
| Texture | NG and NS | NG and NS | NG and NS | NG and NS |
| Melting point °C | 60–63 | 61–62 | 60–64 | 60–62 |
| Breaking point (grams) | 140 | 160 | 150 | 140 |
| Solubility test | Chloroform | Chloroform | Chloroform | Chloroform |
| Hardness (kg/cm²) | 1.5 | 1 | 1 | 0.5 |
| pH | 6.3 | 6.4 | 6.5 | 6.5 |
| Aging stability | Smooth | Slightly rough | Smooth | Bleeding defect |
| Spreadability | Intermediate | Intermediate | Intermediate | Intermediate |
| Surface anomalies | No defects | No defects | No defects | No defects |

NG: No Gritty, NS: No Grease

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