

**Short Communication**

**FORMULATION AND EVALUATION OF HERBAL LIPSTICKS FROM CARROT (*DAUCUS CAROTA L*) EXTRACT**

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Received: 02 Dec 2015 Revised and Accepted: 03 Feb 2016

**ABSTRACT**

**Objective:** To prepare and evaluate natural lipsticks made from the standardized extract of carotenoid of carrots (*Daucus carota L*) with a variety of castor oil base and an irritation test.

**Methods:** The shade-dried coarsely powdered carrot was extracted with ethanol. The extract was subsequently standardized based on the Indonesia's herbal pharmacopoeia. Castor oil was later added as oil base with 50%, 60%, and 70% concentration. Next, lipsticks were evaluated for their physical properties and hedonic test. The open patch test analyzed by skin response was described based on the formula examination and interpreted according to the criteria established by the International Contact Dermatitis Research Group.

**Results:** The results showed that all the lipsticks were stable and had a good force of application while the breaking point reached 76.67–106.67 g. The melting points of the lipsticks containing 50%, 60%, and 70% castor oil were 56, 55, 53.5 °C respectively, while the pH test resulted in 4.4, 4.7, and 5.2. In addition, the hedonic test showed that respondents liked the exciting color, fragrant smell, and oily texture of the lipsticks. The lipsticks themselves did not cause any irritation, so they were safe to wear.

**Conclusion:** The lipstick formulations had met the physical requirements, stability standard, as well as a safety requirement.

**Keywords:** Lipstick, Carotenoid, Carrot, *Daucus carota L*, Castor oil

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Cosmetics with natural ingredients are believed to be safer than chemical-based cosmetics; in addition, natural cosmetics have the ability to protect the skin from Ultra Violet rays [1, 2]. One of the widely used cosmetics nowadays is lip color in the form of crayon or better known as lipstick [3], which can also serve as both natural and chemical.

The development of natural cosmetic ingredients must be accompanied by efforts to improve the quality of raw materials used in the extracts. Due to the many variables that influence the amount and content of active ingredients in a plant, raw materials are not guaranteed to have homogeneous active constituents qualitatively and quantitatively. Therefore, standardization of raw material extracts of natural sources is demanded from researchers in the field of herbal cosmetics [2].

One of the natural sources for cosmetics is a carrot (*Daucus carota L*) [4]. It is a fruit or vegetable containing a large amount of carotenoid compound, ranging from 0.006 to 0.0548 g/100 g [5]. The high carotenoid content can be used as a natural dye, and the carotene itself can act as a precursor of vitamin A, so it adds value to carrots [1, 4-5]. Products using natural coloring, such as carotenoid, are getting more popular as the public becomes more concerned about the long-term effects of synthetic materials.

Cosmetic products, especially lipsticks as they are applied to the lips of a sensitive nature, need to pass an irritation test [3, 6-7]. This should apply to carrots as well because although numerous studies found that carrots are safe [8], a safety test is still required to ensure the possibility of an allergic response that can appear on the skin, including the lips [9].

Therefore, this research aimed to formulate and evaluate herbal lipsticks made from the standardized carotenoid extract of carrots (*Daucus carota L*) with castor oil base variations completed with an irritation test.

Fresh carrots were harvested from a local market in Magelang Regency, Central Java, Indonesia. The Carrots were further dried in an oven at 40 °C. The sample was coarsely powdered using a mixer grinder and stored in an airtight container.

All analytical grade chemicals, acids and solvents, media and other chemicals used in this study were recruited from different sources. They included castor oil, cera alba, lanolin, paraffin wax, vaseline alba, cetyl alcohol, oleum rosae, methylparaben, Butyl Hydroxy Toluene, toluene, hydrochloric acid, Pb (pro analysis), cadmium, a plate of silica gel GF 254, acetic acid, acetonitrile, acetic acid, and  $\beta$ -carotene standards. For the metal content analysis, an Atomic Absorption Spectroscopy (Perkin-Elmer 5100 PC) was used.

50 g of the powdered air-dried sample was mixed with 450 ml of ethanol 96%. The mixture was subsequently incubated for 24 h and filtered. Next, the solvent was evaporated under vacuum, and the resulted extract was kept at 4 °C.

The moisture content was determined with a specific procedure. The extract was placed in an aluminum moisture dish and dried to a constant weight in an oven at 100–105 °C [10]. The weight loss of the sample was then calculated using the following formula:

$$\text{moisture content} = \frac{\text{weight loss}}{\text{weight of sample}} \times 100$$

Total ash content was determined with a specific procedure. The extract was accurately weighed in a previously ignited and tared crucible. The material was then spread in an even layer and ignited by gradually increasing the heat to 600 °C in a muffle furnace until it was white, indicating the absence of carbon. The crucible was cooled in a desiccator, and it was weighed afterward [10]. The content of total ash in the dried material was calculated as:

$$\text{Total Ash} = \frac{\text{Total ash weight}}{\text{weight of sample}} \times 100$$

Acid-insoluble ash content was determined with a specific procedure. The ash derived from the determination of total ash content was boiled using 5 ml of nitric acid for 15 min. After that, the acid-insoluble residue was collected, filtered through a sintered glass crucible as well as ashless filter paper, and rinsed using hot water. Next, the result was ignited to reach a constant weight and weighed. The acid-insoluble ash content was then calculated towards the air-dried extract [10].

$$\% \text{ acid insoluble Ash} = \frac{\text{Acid Insoluble Ash Weight}}{\text{weight of sample}} \times 100$$

The amount of sodium and potassium presenting in 1 g of plant material was estimated by a flame photometry. A number of other metals were calculated using an Atomic Absorption Spectroscopy (SpectraAA-40).

The extracts were subject to physical examination of color, odor, taste, and texture for the organoleptic test. The total carotenoid

content was determined using a thin-layer chromatography with  $\beta$ -carotene standards.

The herbal lipsticks were formulated based on a previously referred method [11]. Lipsticks were prepared from the standardized extract of carotenoid from carrots (*Daucus carota* L) with a variety of castor oil base as an F1 (first formula), F2 (second formula) and F3 (third formula). The ingredients used in the formulation of the lipsticks are given in table 1.

**Table 1: Formulation of herbal lipsticks**

Ingredients (g)	F1	FII	FIII
Castor oil	50.0	60.0	70.0
Vaseline alba	5.0	5.00	5.00
Cera alba	7.50	7.50	7.50
Lanolin	2.50	2.50	2.50
Carrot extract	13.5	13.5	13.5
Cetyl alcohol	4.92	4.92	4.92
Paraffin wax	2.50	2.50	2.50
Oleum rosae	0.50	0.50	0.50
Methylparaben	0.06	0.06	0.06
Butil Hidroksi Toluena	0.06	0.06	0.06

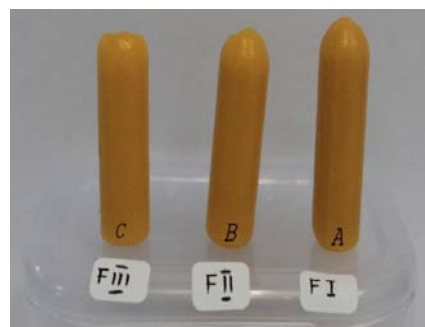
The pH of various formulations was determined using a Digital pH meter. 1 g of lipstick was dissolved in 100 mL of distilled water and measured for its pH [12]. The breaking point test was carried out to determine the lipsticks' strength. Lipstick was held horizontally in a socket ½ inch away from the edge of the support. The weight was gradually increased by a specific value (10 g) at a specific interval of 30 seconds, and the weight at which it could break up was considered as the breaking point. The breaking point of formulated herbal lipsticks was found to be 30 g at 90 s [11]. A piece of coarse brown paper was kept on a shadowgraph balance, and lipstick was applied at 45° angle to cover fully a 1 square-inch area. The pressure reads an indication of the force of application [13]. The melting point of lipstick was determined by melting 50 mg of the sample lipstick, putting it in a glass capillary and cooling it in an ice bath for 2 h. The above glass capillary was fastened with a thermometer, and the whole set up was dipped into a beaker full of water. The water was heated with continuous stirring, where the temperature at which the sample moved into the water was noted as the melting point of lipstick [12].

The hedonic test was conducted by asking 15 respondents to complete a questionnaire related to the odor, color, and texture of the lipsticks [14]. The formulated herbal lipsticks were tested after 20 d to record the fragrance [12]. Patches (materials) were applied to the skin of the back with an occlusive dressing and left undisturbed for a 24-h period. For the irritation potential, a panel size of 10 individuals was utilized [7].

Prior to the research, the ethical clearance KE/FK/529/EC has been presented to the Medical and Health Research Ethics Committee (MHREC) in the Faculty of Medicine, University of Gadjah Mada. The study also identified the macroscopic properties of carrot plants in the Laboratory of Pharmaceutical Biology the Islamic University of Indonesia to match the morphological state of leaves, fruits and roots of carrot plants with the determination keys instructed in the Flora of Java literature.

The results revealed that the moisture content of carotenoid extract from carrots (*Daucus carota* L) was  $23.8 \pm 0.1\%$ . This result was similar to the other research [15]. Excess moisture can result in the breakdown of important constituents by enzymatic activity, which may encourage the growth of yeast and fungi during storage. The total ash values highly indicated the presence of more siliceous matter and calcium oxalate crystal. According to the results, the total ash of carotenoid extract from carrots (*Daucus carota* L) was  $3.34 \pm 0.1\%$ . Meanwhile, the acid-insoluble ash was used to measure the surface contamination of vegetables and fruit. In this study, the acid insoluble ash content was  $1.13 \pm 0.1\%$ . The elemental content of cadmium and lead was also analyzed in the extract, and the results showed the absence of heavy metals.

In the meantime, the extracts showed a brownish orange color, a typical carrot odor, sweetish taste, and a thick texture. The total carotenoid content of the extracts from carrots (*Daucus carota* L) was  $157 \mu\text{g}/100 \text{ g}$  extract. In the organoleptic test, the characteristics of each lipstick formulation were identified. In Formula I (50% castor oil), the lipstick structure was loud and rather greasy, Formula II (castor oil 60%) resulted in a hard and greasy lipstick while Formula III (70% castor oil) produced a more oily and mushy one. The smell of each formula was the same as they all contained oleum rosae. Meanwhile, the color of the three formulas was almost the invariable, orange. Therefore, the differences in the level of castor oil only influenced the structure of the lipsticks.



**Fig. 1: Lipstick formulations from carrot extract with 50% (A), 60% (B), and 70% (C) of castor oil content**

All the fabricated lipsticks were then evaluated for their pH, breaking point, force of the application, melting point, hedonic test, perfume stability, and skin irritation (table 2).

The test results showed that the pH of formula I, II, and III reached 4.4, 4.7, and 5.2 respectively due to the discrete levels of castor oil in each formula. Castor oil is alkaline, so the higher level of castor oil results in a more alkaline lipstick.

A breaking point shows the ability of lipstick to encounter a mechanical process. A high level of castor oil gives a lower value of breaking point. As a result, the addition of castor oil in a large quantity causes the lipstick to become mushy.

The force of application test is designed to measure the force applied to the formula comparatively. The herbal lipsticks in this research indicated a good result of the force of the application test.

Table 2: Evaluation of herbal lipsticks (F1, F2, and F3)

Evaluation parameters	F1 (50%)	F2 (60%)	F3 (70%)
pH	4.4	4.7	5.2
Breaking point	106.67+5.74	96.67+5.74	76.67+10.6
Force of application	Good	Good	Good
Melting point	56 °C	55 °C	53.5 °C
Hedonic test	Good Response	Good Response	Good Response
Perfume stability	stable	stable	Stable
Irritation open patch test	No irritation	No irritation	No irritation

A perfect lipstick has a high melting point (>50 °C) in order not to melt easily in the heat, and it has to be firm enough to withstand pressure when applied. However, it must also be soft and easy to apply, spread evenly, and form a spongy, attractive film [12]. The melting point test in this study showed that the more castor oil was used, the lower the melting point was.

Meanwhile, the hedonic test showed that the respondents liked the lipsticks' exciting color, fragrant smell, and oily texture. This type of test is important to assess consumers' interest in herbal lipsticks. The test also found that the perfume stability test on Formula I, II, and III showed good odor stability during the 20 d of storage.

A patch test is useful in identifying the types of reactions to a particular lipstick, whether it is irritant or allergic. The standard test series can help in identifying the agents causing irritation. Of all the formulas, no irritation to the skin was found when given herbal lipsticks. This means that these herbal lipsticks were safe to wear. Previous research suggests that the use of herbs as dye lipstick will not give side effects [16].

The research results showed that all the lipsticks were stable, had a good force of application. In addition, the hedonic test showed that respondent loved the exciting color, fragrant smell, and oily texture of the lipsticks. Finally yet importantly, lipsticks did not cause irritation, so they were safe to apply.

#### ACKNOWLEDGEMENT

The authors are grateful to the Directorate of Research and Community Service of the Islamic University of Indonesia for providing the facility and funding to carry out this research.

#### CONFLICT OF INTERESTS

All authors have none to declare

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