Bulletin of Environment, Pharmacology and Life Sciences
Bull. Env. Pharmacol. Life Sci., Vol 10 [5] April 2021 : 140-145

Online ISSN 2277-1808
Journal's URL:http://www.bepls.com
CODEN: BEPLAD

ORIGINAL ARTICLE **OPEN ACCESS**

Formulation and Evaluation of Lipsticks from the Dyes Of Root Barks of *Alkana tinctoria*

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ABSTRACT
Alkana tinctoria (Family: Boraginaceae) is a herb indeginous to the mediterranian region. The study aims at separation of several reddish pigments from the root barks of this plant and incorporation into lipsticks. The phytoconstituents were extracted initailly using petroleum ether followed by solubilizing the concentrated extract in methanol. Two types viz. methanol soluble and methanol insoluble dyes were separated. Lipsticks were formulated using various concentration of the extracted dyes. Several organoleptic and physico-chemical parameters such as color, odor, size, color of applied area, dimensions, water resistance, load test, softening point and melting points were evaluated. Moreover, TLC and HPTLC were performed using Petroleum ether: Ethyl acetate: Glacial Acetic acid (8:1.5:0.1) and Methanol: water (6.5:3.5) as mobile phase. Alkannin was found in methanol soluble fraction. Methanol insoluble components were able to impart concentration dependent darker shades to the lipsticks in comparison with the methanol soluble components. Moreover, lipsticks formulated of methanol soluble fraction exhibited more durability (in terms of breaking point). The methanol soluble fraction of petroleum ether extract of root bark of Alkana tinctoria can be effectively incorporated for the development of herbal lipsticks or colorants.
***Keywords**: Alkana tinctoria, ratanjot, alkannin, lipsticks.*

Received 02.01.2021 Revised 20.03.2021 Accepted 31.03.2021

INTRODUCTION

Lipsticks are an age-old formulation that occupies irreplaceable position in the arena of cosmetics. Commercially liptics are avialable in various types *viz* Matte, Shimmer, Metallic, Frost, Glossy, Satin, , etc. Despite their conventional application of protection, enhancing aesthetic appearance and masking the defects; lipsticks nowadays enjoy the privileges of cosmeceuticals [1].

However, scientific reports have underlined the carcinogenicity, allergic contact dermatitis and skin toxicities due synthetic colorants used in the commercial lipsticks[2, 3]. Nevertheless, high cost and meagre availability of luxurious brands have added to the sales of pirated or misbranded product with more carcinogenic colorants.

Alkana tinctoria is a herb of Boraginaceae family [4]. It is indegenous to mediterranena region and commercially known as ratan jot or dyers alkanet [5]. However, is prominently found in Himalayan region [6, 7].

The root bark contains various reddish oil soluble pigments. Since, ages the root bark was used in culinary preparation such as *rogan-josh* etc or in topical oil formulation in order to impart red color. Moreover, the bark also has several health benefits as it is antimicrobial [8],anthelmintic and antiinflammatory [7] antioxidant properties [10]. The tincture of its roots is commonly utilized in microscopy and preliminary phytochemical analysis for testing oils, lipids and fats.

The current study is designed considering the lipophilic nature of these pigments [11]. Efforts are made to incorporate the red pigments in lipsticks, as the lipstic base is completely lipophilic. Various concentrations of different dyes are incorporated and the sticks are evaluated.

MATERIAL AND METHODS

Alkana tinctoria root barks were obtained from local market of Pimpri-Chinchwad region in Pune.Petroleum ether (b.p. 60-80 ^{0}C) and methanol was purchased from Merck Life Sciences Pvt. Ltd., Mumbai. Beeswax, parraffin oil were purchased from Yucca Enterprises Mumbai.

The *Alkana tinctoria* root barks were air dried for 6 hrs and crushed with hands to abtain coarse powder. The powder was sifted though 80# seive inorder to separate fine powder. The fine powder was discarde as it clogs the pores of filter as well may enter in the final extract. The coarse powder was extracted with petroleum ether (b.p. 60-80 ^{0}C) in a soxhlet apparatus for more than 4 hours until colorless liquid was obtained in the siphon tube.

The petroleum ether extract was concentrated *invacuuo* and cooled. Later, the cooled semisolid extract was shaken with methanol for 3 hrs on a rotary orbital shaker and filtered to separate methanol soluble fraction. The residue (methanol insoluble fraction) and filtrate were used for the formulation of lipsticks.

The methanol soluble fraction was found to be less darker in shade compared to the methanol insoluble one. Both the fractions were subject to chromatographic separation to obtain the intense and dark pigments. Preparative thin layer chromatography was performed using silica gel G as stationary phase and two different mobile phases Petroleum ether: Ethyl acetate: Glacial Acetic acid (8:1.5:0.1) and Methanol: water (6.5:3.5) were employed to separate darker color pigments from both the fractions. Similarly, HPTLC was performed on using sample applicator (CAMAG-Linomat 5) and detection was made in both visible and UV 365 using (CAMAG TLC scanner-3).

Lipsticks were formulated using molten beeswax, white soft paraffin, fractions of *Alkana tinctoria* and perfume. The molten mixture was added in the wells of pre-cooled stainless steel molds which were internally coated with white soft paraffin. The two fractions were employed for formulation of lipsticks. Initially colorless lipsticks were formulated using various proportion of beezwax, white soft paraffin and perfume were prepared to determine the weight for calculation of the concentration of dye to be used. The proportion of beeswax and white soft paraffin (2: 1) was found to be effective. The methanol soluble fraction was used in various concentrations (0.25, 0.5, 1, 2 and 3 mg) to develop range of shades. These lipstick batches were named A, B, C, D and E respectively. Similarly, methanol insoluble fraction was used in varying concentration of 0.5, 1, 2 and 3 mg to develop batches F, G, H and I respectively.

Various physico-chemical parameters recorded were color, odor, size, water resistance, breaking point, softening point, melting point and color of applied area. Color was observed by placing the lipsticks on white background and observing under fluorescent white light. Odour was recorded by direct sniffing the lipsticks as well as applied area. Dimensions i.e length and diameter were measured using vernier calliper.

The performance charateristics such as water resistance of the applied area was recorded by applying a thin uniform coat over a tissue paper and by adding 5-6 drops of water with the help of pipette. Prevention of wettability of the paper was considered as water resistance.

Breaking point was determined for each type lipsticks. Load in the form of water was added dropwise to a beaker suspended with copper wire to the centre of lipstic, untill the lipstic breaks. Later, the water in the beaker was weighed to determine the specific load at which the lipstic deforms or breaks. To ascertain the softening point, the lipsticks were maintained at an environmet with gradual rise in temperature (1 ^{0}C/min). The point at which the lipstic softens was recorded. Melting point was performed using digital melting point apparatus. The lipsticks were separately grated and added in a narrow tube and the tube was placed in silicone oil for recording the melting range.

RESULTS AND DISCUSSION

Application of cosmetics drastically alter the appearance and hence improve the gorgeousness therefore it is considered as an attractiveness enhancer. Moreover, make-up are also applied to elevate the self-esteem level by promoting the physical allure and also to provide a feel-good effect in the stressful times [12].

The current market is laden with synthetic colorants not only in cosmetics but also in edibles. Most of the synthetic colorants employed in cosmetics have a history of carcinogenicity. Moreover, the piracy of cosmetics in developing and underdeveloped countries remains a major issue. Herbal dyes have enormously benefitted the mankind since ancient times.

Alkana tinctoria have been scientifically proven to have numerous health benefits *viz* antioxidant [13], antimicrobial [14], anthelmintic and antiinflammatory [9].

The yield of petroleum ether (60-80 ^{0}C) extract of *Alkana tinctoria* was found to be 7.44 % w/w. Further, the methanol soluble and methanol insoluble fraction was observed to be 35 % and 65 % w/w respectively of the petroleum ether extract.

Upon screening the fractions for HPTLC, it was observed that the methanol soluble fraction contained significant amount of Alkannin using methanol: water (6.5: 3.5) as solvent system. (Fig 1) Our findings are concurrent with the earlier reports [4]. Thus, the red color imparted to the lipsticks a, b, c, d and e (Fig 2) was due to the presence of Alkannin.

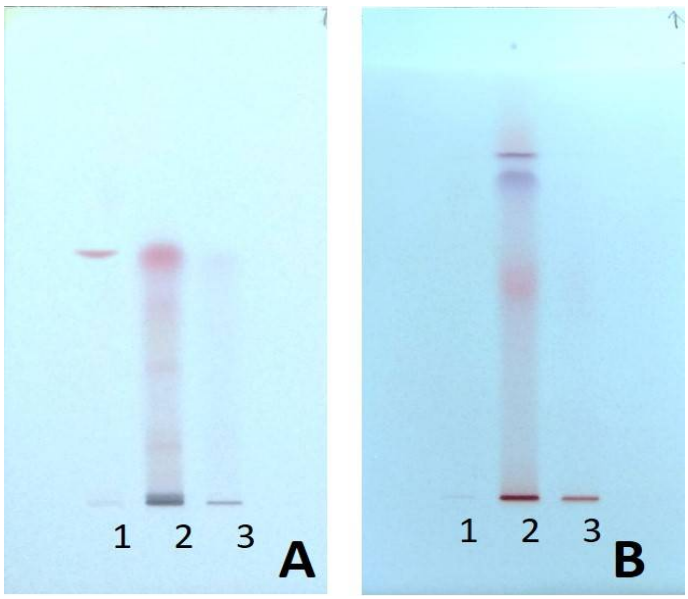

Fig 1 HPTLC Chromatograph Plate A: Solvent system methanol: water (6.5: 3.5); Plate B: Solvent system Petroleum ether: Ethyl acetate: Glacial Acetic acid (8:1.5:0.1). Spot 1 standard Alkannin, Spot 2 Methanol soluble fraction and Spot 3 Methanol insoluble fraction.

The color of the lipsticks was found to be ranging between reddish-pink to reddish brown depending upon the concentration of the extract.The reddish color is the characteristic of *Alkana tinctoria* phytoconstituents. Alkannin the major alkaloid along with pseudo-tanninsimpart reddish coloration to the formulation [8,11]. However, reddish color of the lipsticks formulated using petroleum ether extract could be attributed to the presence of Alkaloids, flavonoids, steroids resins and bufadenolides [8]. (Fig 2)Rose oil 0.1 ml was added to eachlipstick in order to mask the odor of *A. tinctoria* extract. Therefore, the odor was pleasant rose like.

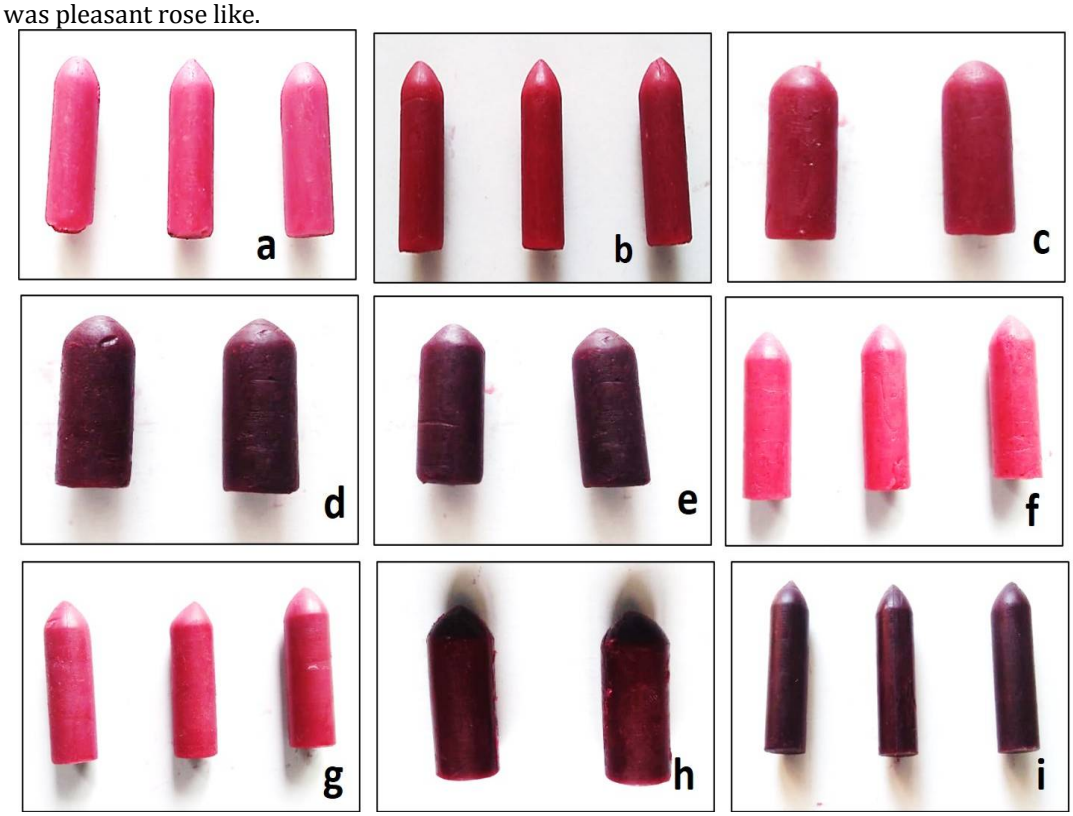

Fig 2: Effect of various concentrations of pigments on the colour shades of formulated lipsticks. a, b, c, d and e contain 0.25, 0.50, 1.0, 2.0 and 3 mg respectively of methanol soluble pigments. f, g, h and i contain 0.50, 1.0, 2.0 and 3 mg respectively of methanol insoluble pigments.

Dimensions of the lipsticks were measured with the help of vernier caliper. All the lipsticks were found to have almost similar dimensions as all were produced from same molds. (Fig 3) The color of applied area, water resistance evenness and gloss were observed and recorded at qualitative parameters and were graded on a 5-point scale.

Fig 3: Dimension of lipsticks

The color of applied area was found to be even, the gloss quality and water resistance for lipsticks containing 0.25, 0.5 and 1 mg of pigment was observed to be significantly higher compared to the lipsticks formulated using 2 & 3 mg of pigment that resulted in darker shade however, poor coat uniformity (Fig 4), gloss and water resistance(Fig 5). Thus, it can be observed that the excess pigment concentration changes the physicochemical properties of the lipsticks.

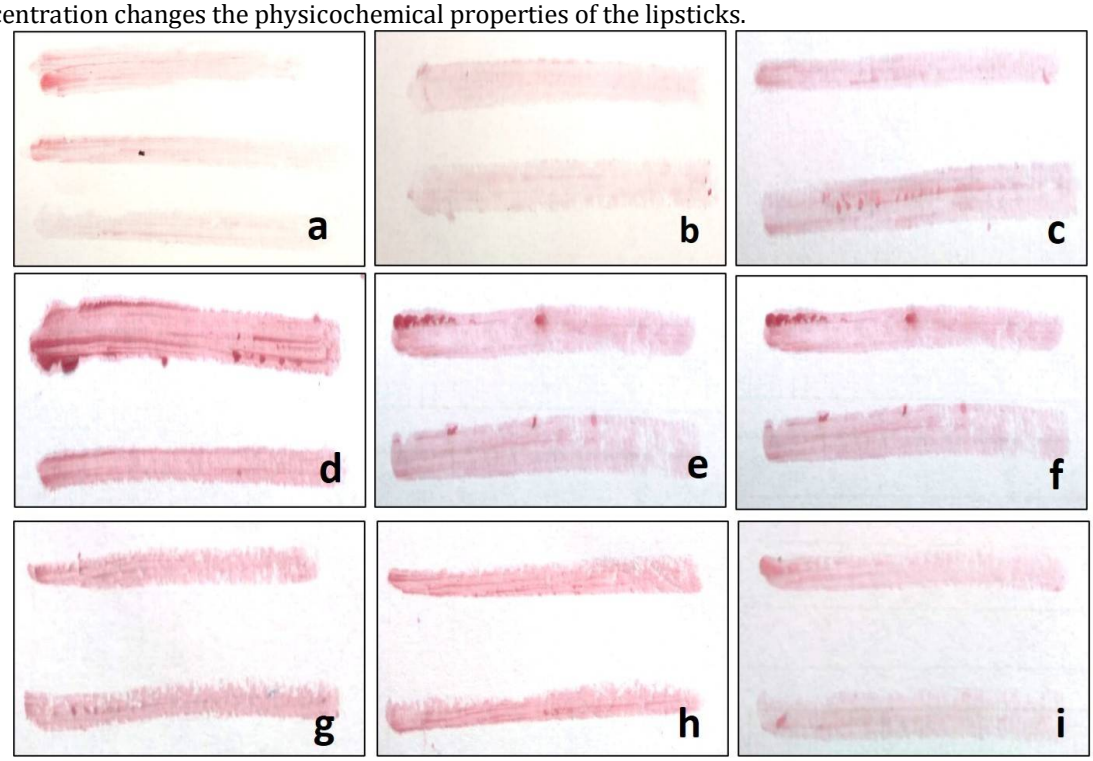

Fig 4 Coating imparted by the lipsticks on the rough surface. a, b, c, d and e contain 0.25, 0.50, 1.0, 2.0 and 3 mg respectively of methanol soluble pigments. f, g, h and i contain 0.50, 1.0, 2.0 and 3 mg respectively of methanol insoluble pigments

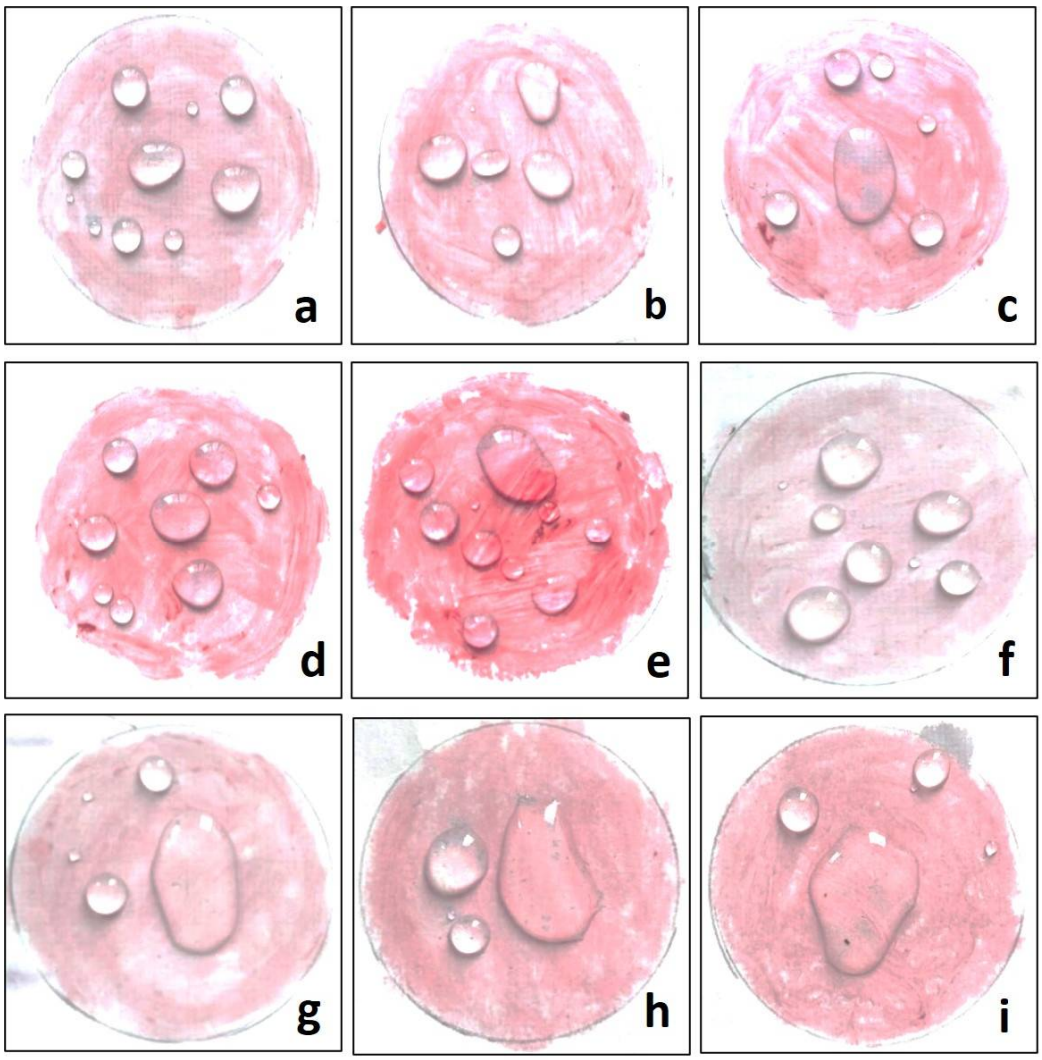

Fig 5 Water resistance of the formulated lipsticks. a, b, c, d and e contain 0.25, 0.50, 1.0, 2.0 and 3 mg respectively of methanol soluble pigments. f, g, h and i contain 0.50, 1.0, 2.0 and 3 mg respectively of methanol insoluble pigments

It is also evident from the data obtained from breaking, softening and melting points. Table 1and 2 depicts the findings of various parameters of lipsticks prepared using methanol soluble and methanol insoluble pigments respectively. All values are expressed as mean ± SEM (n=6). It is evident from our findings that the lipsticks prepared using methanol soluble pigment at a concentration of 2 mg resulted in superior quality of lipsticks compared to the rest. However, the breaking point of these lipsticks were found to be poor. Thus, there arise a need for implementation of a hardening agent that can provide stiffness to the lipsticks without affecting its coating ability.

Table 1: Physico-chemical parameters of lipsticks formulated using methanol soluble pigments

Parameters	A	B	C	D	E
Color	Light Red	Red	Dark Red	Light Brown	Brown
Odor	Rose like	Rose like	Rose like	Rose like	Rose like
Size in mm	L = 35 ± 1 W = 11 ± 0.4	L = 35 ± 0.9 W = 11 ± 0.4	L = 35 ± 0.9 W = 11 ± 0.3	L = 35 ± 1 W = 11 ± 0.5	L = 35 ± 1 W = 11 ± 0.3
Breaking Point (load in gm)	51.8 ± 1.3	49.6 ± 1.0	47.3 ± 1.5	40 ± 1.0	63.7 ± 1.4
Softening Point (^{0}C)	52 ± 1	51 ± 2	48 ± 1	45 ± 2	55 ± 1
Melting range (^{0}C)	64 - 65	61 – 62	59 - 60	53 – 56	68 - 70

L= length, W = width, All the values are expressed as Mean ± SEM, (n=6)

Lipsticks A, B, C, D and E contains 0.25, 0.50, 1.0, 2.0 and 3 mg respectively of methanol soluble pigments

Table 2: Physico-chemical parameters of lipsticks formulated using methanol insoluble pigments

Parameters	F	G	H	I
Color	Pink	Red	Light Brown	Brown
Odor	Rose like	Rose like	Rose like	Rose like
Size in mm	L = 35 ± 1.1 W = 11 ± 0.5	L = 35 ± 1.0 W = 11 ± 0.4	L = 35 ± 0.8 W = 11 ± 0.3	L = 35 ± 0.9 W = 11 ± 0.5
Breaking Point (load in gm)	80.2 ± 1.2	50 ± 1.3	110.4 ± 2.2	173 ± 2.7
Softening Point (^{0}C)	40 ± 1	44 ± 1	46 ± 2	45 ± 1
Melting range (^{0}C)	45 - 48	51 – 53	52 - 55	53 - 55

L= length, W = width

Lipsticks F, G, H and I contained 0.50, 1.0, 2.0 and 3 mg respectively of methanol insoluble pigments

CONCLUSION

The study proves that better quality of lipsticks can be formulated by the use of various phytoconstituents of *Alkana tinctoria*. Demand for vegan and cruelty free cosmetics is surging and, in these times, lipsticks developed from herbal sources can prove to be beneficial not only for the consumer but also for the producers.

ACKNOWLEDGEMENT

The authors would like to extend their gratitude towards Progressive Education society's Modern College of Pharmacy, Nigdi, Pune 411044 for providing necessary facilities and infrastructure for the research work.

COMPETING INTEREST

The authors have declared that no competing interest exists

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CITATION OF THIS ARTICLE

B. P. Pimple, Mekhe A.R., Joshi A.A., Malwadkar S.S. and Pimple K. B. Formulation and Evaluation of Lipsticks from the Dyes of Root Barks of *Alkana tinctoria*. Bull. Env. Pharmacol. Life Sci., Vol 10[5] April 2021: 140-145.