Published online: 20 July 2018

Development of Liquid Lip Product Containing Dragon Fruit Skin Extract

Melissa Sinsiriphan
Email: malimaeeeee@gmail.com
Master of Science in Cosmetic Science
School of Cosmetic Science Mae Fah Luang University
Ampa Jimtaisong
Email: ampa@mfu.ac.th
School of Cosmetic Science Mae Fah Luang University

Abstract

This research aimed to extract and stabilize dragon fruit skin extract as well as to incorporate the extract in lip tint formulation as it is a non-edible part and often a waste. However, red color extracted from dragon fruit skin was not as intense as expected and unstable under extreme conditions, 45^{o}C and fluorescence light exposures. The stability test results have shown that dragon fruit skin extract encapsulated with alginate has better stability than free pigment. The ΔE* value of encapsulated extract was 77.71% lower than that of free color extract by the end of week 4. In addition, both free and encapsulated extracts incorporated in clear lip tint formulation provided pinkish red color to the formulation but could not stain skin. For this reason, synthetic color was needed to be added in order to achieve red stain on skin. According to the moisturizing effect test on 10 volunteers, lip tint formulation with 2.5% free dragon fruit skin extract gained the highest average percentage moisture increase of 49.04±13.76% with the p-value of 0.000 comparing with that containing the encapsulated extract. Therefore, dragon fruit skin extract is more preferred to be used as a moisturizing agent rather than a colorant.

Keywords: Dragon Fruit Skin/Lip Tint/Natural Colorant/Moisturizing

Introduction

As an ecological trend is emerging, consumers pay more attention on natural cosmetics that are not only functional, but also have good appearance. Dragon fruit skin

is often a waste as it is nonedible part. There has been shown that extract from dragon fruit skin possessed antioxidant activity and betalain that gives red color. In addition, Phebe et al. (2009) and Harivaindaram et al. (2008) had suggested the use of dragon fruit skin as a natural colorant, thickening agent or as a moisturizing agent in cosmetic products (Stintzing et al., 2002). However, natural colors are generally less stable comparing to synthetic colors. Santos et al. (2013) have shown a successful result in using alginate as an encapsulation material for anthocyanin stabilization. As a result, applying alginate to encapsulate betalain from dragon fruit skin might be possible. Lip tint is one of the popular cosmetic products in Thailand and comes in different colors and texture. Lip tint products are usually water soluble and compatible with water-soluble substances. Hence, to involve dragon fruit skin extract in lip tint product, would confirm the efficacy and the usability of the fruit skin itself.

Dragon fruit has different variations of color known as white, magenta, red and yellow (Juarez et al., 2010). The coloring application of the dragon fruit originates from its betalain pigments. Betalain is a class of natural pigments including yellow to orange betaxanthins and red to violet betacyanins. Betalains in the dragon fruit pulp and peel were reported to be identical (Wybraniec et al., 2001). In addition, stability of betacyanin is affected by heat, oxygen, light, pH and moisture (Woo et al., 2011). Woo et al. (2011) have found that the best condition in preserving betalain pigment in red dragon fruit juice is pH 5.0 at 4°C. In addition, Jamilah et al. (2011) have reported that 10.79% of pectin was found in dragon fruit skin. Pectin is a non-starch water-soluble linear polysaccharide found in higher plants, particularly in fruits and vegetables. The naturally derived polymers are hydrated in aqueous environment and create a hydrogel. This system is imparting moisturizing effect which water is immobilized by insoluble polymers (Kanlayavattankul & Lourith, 2015). Therefore, applying dragon fruit skin extract as a moisturizing agent in lip tint formulation might be possible.

According to the reviewed information, extraction and encapsulation of dragon fruit skin extracts with calcium alginate were studied in this work. Moreover, stability of free and encapsulated extracts, as well as lip tint formulations with free and encapsulated extracts were investigated. Finally, efficacy test and moisture effect of the finished product was conducted.

Research objectives

1. To stabilize dragon fruit skin extract by encapsulation method
2. To study the stability of natural colorant derived from dragon fruit skin
3. To measure the skin moisturizing by using dragon fruit skin extract in lip tint formulation

Research methodology

1. Preparation of dragon fruit skin

Dragon fruit skins were cut into small pieces (approximately 1 cm width and 3 cm length) and put on tray before putting into 50^{o}C hot air oven for 4 hours. The completely dried skins were taken out and blended using blender in order to achieve powder form.

2. Color extraction from dragon fruit skin powder

150 g of dried dragon fruit skin powder was soaked in 800 ml of water solution of 1% acetic acid for 1 hour using magnetic stirrer at 50 rpm. After that, residue was filtered using booster machine. Deep pink color solution was taken to evaporate using rotary evaporator at 45^{o}C.

3. Encapsulation of color pigment with calcium alginate

The ratio between calcium chloride and sodium alginate was varied in order to get the desired alginate beads which are easy to break and have the least residue when rub on skin. After that, the extracted color was encapsulated using selected ratio of calcium chloride and sodium alginate and varied at different percentages to get desirable beads which are darkest pink color.

4. Stability test of free pigment and encapsulated pigment

4.1 Thermal stability test

Free and encapsulated color extracts were put into clear glass bottles and wrapped with foil paper before storing in 4^{o}C, 25^{o}C and 45^{o}C conditions for 30 days cycle. Color changes were observed by using chromameter for measurement. The results were reported in L* a* b* values on weekly basis.

4.2 Fluorescence Light sensitivity test

Free and encapsulated color extracts were put into clear glass bottles and stored under dark and fluorescence conditions at 25^{o}C. Color changes were observed

by using chromameter for measurement. The results were reported in L* a* b* values on weekly basis.

5. Lip tint formulation

Five lip tint formulations were formulated using water as a main ingredient. Humectants, glycerin and hydroxyethyl urea, were added to increase water retention for lips. Carbopol ultrez-21, Acrylate/C10-30 alkyl acrylate crosspolymer was used as a gelling agent in the formulation. Combination of preservatives was used to enhance the lip tint shelf-life. Clear lip tint formulation (F1) was formulated to be used in finished product stability test while other 4 formulations were to be used in moisturizing effect test.

6. Stability test of finished product

6.1 Thermal stability test

Lip tint product with free and encapsulated color extracts were put into clear glass bottles and wrapped with foil paper before storing in 4^{o}C, 25^{o}C and 45^{o}C conditions for 30 days cycle. Color changes were observed by using chromameter for measurement. The results were reported in L* a* b* values on weekly basis.

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7. Moisturizing effect test

Lip tint products with free and encapsulated extract were tested on armrest of 10 volunteers using hydrometer (Moritex Moistsense, Japan) to measure moisturizing effect scoring from 0-99.

Results and discussion

1. Preparation of dragon fruit skin

Dragon fruit skins were put in 50^{o}C hot air ovens for 4 hours. The skins were dried and red. After the skins were blended with blender, dried skins turned into powder with a little bit lighter red color with 20% yield.

2. Color extraction from dragon fruit skin powder

The powder of dragon fruit was extracted using of water solution of 1% acetic acid with the pH of 2.71. After the skin residue was filtered out, the result was a clear red color solution. After that, clear red color solution was evaporated at 45^{o}C to achieve more intense red color. The percentage yield of the crude extract based on the weight of dragon fruit skin powder was 24.20% with the pH of 4.49.

3. Encapsulation of extract with calcium alginate

The most desirable outcome was 1% calcium chloride with 1% sodium alginate which resulted in most easy to break beads and had very small residue when rub on skin. After that, percentage of crude extract was varied and put in 1% sodium alginate solution and dropped into 1% calcium chloride solution using syringe with number 23 needle. The best crude extract percentage for encapsulation was 5% where 5 g of crude extract were put in 95 g of 1% sodium alginate solution. The pH of sodium alginate solution mixed with 5% of crude extract was 4.51. The result was pink beads that were easy to break and had a very small residue when rub on skin. The pink color beads pH was 4.17 (Figure 1).

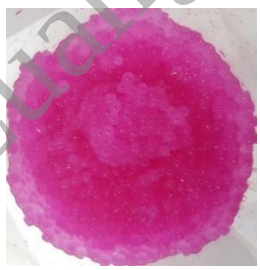

Figure 1 Color beads with 5% of color extract

4. Stability test of free and encapsulated extract

Free color extract was degraded when exposed to high temperatures resulting in lighter color as L* value increased. L* represents the lightness ranging from 0 to 100 where 100 is the brightest (Table 1). The initial dark pink shade of free color extract under 45^{o}C also turned brown within 1 week leading to decreasing a* and increasing b*. a* represents green-red component where green is in negative direction and red is in positive direction. Additionally, b* represents blue-yellow component, with blue in the negative direction, and yellow in the positive direction. For sample that was kept under 4^{o}C was the most stable as ΔE^* values were lower when compared with other conditions week by week. When compare L* values of sample under 4^{o}C between week

0 and week 1, p-value of 0.004 was obtained where other conditions possessed lower p-values of 0.000. This result corresponded with Woo et al. (2011) research that the best condition in preserving pigment in red dragon fruit juice is at 4ºC. In addition, stability of encapsulated color extract was better in every condition comparing with free color extract as shown in lower values of ΔE*. The result was in the same direction with Santos et al. (2013) study in which Ca-alginate encapsulated system was more stable than free extract of anthocyanin.

Free color extract decomposed largely under fluorescence condition resulting in higher value of L* as the color went lighter. In addition, a* value of free color extract under fluorescence light decreased largely comparing with dark condition, indicating fading red color. This outcome correlated with Woo et al. (2011) study that the deterioration of betalain stability was observed when exposed to light. However, when compare L* values of free color extract samples under dark and fluorescence conditions between week 0 and week 1, same p-values of 0.000 were obtained meaning that it would not be able to prevent color deterioration even if samples were stored under dark condition but still exposed to room temperature.

Table 1 L* a* b* measurement of free color extract under 3 conditions

Condition	Week	L*	a*	b*	ΔE
	0	46.58±0.03	13.20±0.02	2.60±0.01	-
4ºC	1	46.99±0.05	8.45±0.04	7.39±0.05	6.76±0.01
	2	50.45±0.10	5.95±0.01	8.95±0.04	10.39±0.06
	3	52.54±0.08	4.42±0.02	9.89±0.02	12.87±0.05
	4	52.01±0.08	2.67±0.02	9.44±0.04	13.56±0.27
25ºC	1	51.23±0.05	3.86±0.01	9.21±0.04	12.35±0.04
	2	52.39±0.05	2.56±0.02	10.01±0.03	14.20±0.01
	3	52.46±0.12	2.05±0.01	10.66±0.10	14.96±0.09
	4	54.87±0.09	1.55±0.06	10.54±0.04	16.35±0.07
45ºC	1	48.15±0.03	2.00±0.01	9.60±0.04	12.47±0.03
	2	49.50±0.06	2.06±0.01	9.72±0.03	13.53±0.04
	3	50.06±0.17	2.30±0.06	10.90±0.09	14.13±0.05
	4	50.41±0.65	2.07±0.02	10.41±0.03	14.13±0.03

However, encapsulated color extraction was less affected in both conditions as its ΔE* values were lower. The result corresponded with Santos et al. (2013) study that

the degradation of encapsulated anthocyanins extract occurred slower than the free extract due to the protection of molecules when encapsulated by the polymeric matrices.

5. Lip tint formulation

Percentages of free color extract and encapsulated color beads were randomly varied and put in lip tint formulation in order to get desirable appearance. Using 50% of free pigment in 50% clear lip tint base delivered the best appearance. However, lip tint was not able to stain skin even though the finished lip tint was dark pink. In addition, using 17% of encapsulated color beads in 83% clear lip tint base resulted in most desirable appearance as the beads were not too tense. However, finished lip tint product was not able to stain skin. In order to add more intense red color to both lip tint formulations, using only free or encapsulated extracts as colorants were not possible. Therefore, 0.2 % of Eurocert Ponceau 4R (Food red 7), synthetic water-soluble color, was added to both formulations.

6. Stability test of finished product

Lip tint formulations with free color extract were eventually faded to lighter color with yellowish tone in every condition. Samples that were put under 25^{o}C and 45^{o}C were faded quickly resulted in highly increasing L* value, decreasing a* value, and increasing b* value. However, encapsulated color beads in lip tint base were gradually changed from light pink to white in every condition. The best condition that encapsulated color beads were slowly degraded was 4^{o}C.

7. Moisturizing effect test

Four formulations of lip tint (F2-F5) were formulated to be used in moisturizing effect test (Table 2, Figure 2). The pH values of each formulations were 6.04, 5.84, 5.72 and 4.53 respectively.

Product samples were prepared and tested on armrest of 10 volunteers using hydrometer (Moritex Moistsense, Japan) as an instrument. The most moisturizing formulation was sample number 3, lip tint with 0.2% synthetic color and 2.5% free extract with the average percentage moisture increase of 49.04±13.76% with the p-value of 0.000 (Table 3). The reason could be because of the polysaccharides in dragon fruit skin that acted as an additional film forming agent giving occlusive effect to skin. Polysaccharides are role by the ability of hydrogel or hydrocolloid mobilizing water to the contact skin (Goddard & Gruber, 1999). Therefore, adding free dragon fruit peel

extract directly in water-based lip formulation would increase moisturizing effect without disturbing the product appearance.

Table 2 Lip tint formulations

Raw material	Sources	F1	F2	F3	F4	F5	Function
DI water	Lee Sia Huad, Thailand	82.86	81.97	79.90	79.90	40.44	Solvent
Methylparaben	Sharon Laboratories, Israel	0.15	0.15	0.15	0.15	0.15	Preservative
Sodium Benzoate	Emerald, USA	0.5	0.5	0.5	0.5	0.5	Preservative
Glycerin	Thai Glycerin, Thailand	6.18	6.17	6.01	6.01	3.09	Humectant
Hydroxyethyl Urea	Akzo Nobel, Singapore	8.23	8.21	8.00	8.00	4.12	Humectant
Carbopol Ultrez-21	Lubrizol, USA	0.76	0.76	0.74	0.74	0.38	Gelling Agent
Triethanolamine	Dow Chemical, Thailand	0.72	0.72	0.70	0.70	0.36	pH Adjuster
Phenoxyethanol	Schulke&Mayr, USA	0.60	0.60	0.60	0.60	0.60	Preservative
Triethanolamine	Dow Chemical, Thailand	0.72	0.72	0.70	0.70	0.36	pH Adjuster
Eurocert Ponceau 4R	Sensient, USA	-	0.20	0.20	0.20	0.10	Colorant
Free color extract	Thailand	-	-	2.50	-	-	
Encapsulated extract	Thailand	-	-	-	2.50	50.00	

F2 F3 F4 F5

Figure 2 The physical appearance of lip tint formulation F2-F5

Table 3 Average percentage of moisture increase tested on 10 volunteers

Sample	Average Percentage Increase (T0-T180)	P-Value (Paired-Sample T Test)
DI Water	1.71±7.48	0.488
F2	25.49±16.76	0.001
F3	49.04±13.76	0.000
F4	34.14±14.82	0.000
F5	32.31±15.55	0.000

However, adding encapsulated extract into lip tint formulation resulted in lower moisturizing scores as the pH values were lower comparing with the formulation with free extract. Lip tint base formulation was formulated using carbopol ultrez-21 as a thickener and film former which is unstable if the pH is below 5. Therefore, adding more encapsulated extract into the formulation would make the lip tint formulation loses its gel texture as pH of the system was more acidic. As a result, the occlusive effect was less as the main film forming agent, carbopol ultrez-21, lost its property.

Conclusion

This research aims to stabilize color extract of dragon fruit skin by using alginate encapsulation method. Nevertheless, the result was not satisfied because the color beads were not able to stain skin when apply and the stability was not good enough. From moisturizing effect test, lip tint formulation containing free extract gave the best result. This could be because of the polysaccharides in dragon fruit skin which performs as an additional skin conditioning agent and film former in lip tint formulation. Therefore, using free dragon fruit skin extract as a moisturizing agent in water-based lip product would be more suitable.

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