

Production of Lip Balm From Stingless Bee Honey

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ABSTRACT *India Cosmetics is used daily by majority of the people worldwide. Nowadays, consumer demand for natural based product cosmetics as they are safe to use and environmentally friendly. Lip balm is a cosmetic or lip care product whose purpose is to prevent dry and chapped lips. The quality of lip balm is directly linked with the basic ingredients used in the formulation. This work involved the formulation of lip balm from natural ingredients. Various composition of beeswax, shea butter, stingless bee honey, oils and colorant were studied to obtain the best formulation. Stingless bee honey was added to the lip balm formulation as moisturizing agent. The physico-chemical properties of the formulations were determined including melting point, stability, moisture content, color intensity, sensory test for human acceptance and microbial test. From the results, the formulation of lip balm from beeswax, shea butter and oil with ratio 1:1:1 was the best formulation. It has high melting point and has stable condition in low and room temperature. The presence of honey in the formulation assisted to increase the moisture content in the lip balm. Besides that, for color intensity, natural colorant from fruit juice and extraction contributed the color to the lip balm but the color intensity was lower compared to powder colorant. Lastly, the formulation of lip balm from beeswax, shea butter and oil with ratio of 1:1:1 met the consumer acceptance as the highest sensory test score was obtained for this formulation.*

Key words: Cosmetic, lip balm, honey, natural ingredients, moisturizing, stability

Introduction

Cosmetics can be defined as substances used to improve or to enhance the appearances of human body (European Commission 1999). Lipstick or lip balms are viewed as one of the most important cosmetic items in women. The essential factors that attract customers are color, smell, ingredients, stability and extensibility of the lipstick. There are few significant differences between lipstick and lip balm. Lipstick give color to the lips while lip balm provides protection to the lips. The main ingredients of lipstick or lip balm are wax, butter, oil, wax, and butter (Kasparaviciene, Savickas et al. 2016). However, the ingredients and composition depends on the type of lip balm to be produced. Commercial lip balm contains synthetic oil and waxes. Ingredients like petrolatum and phthalates can

lead to health problem such as allergies, asthma and cancer (Kadu, Vishwasrao et al. 2015).

A moisturizing lip balm helps to prevent dry and chapped lips by deeply hydrating the outer skin cells of lips (Norazlin 2015). Hence, honey was chosen as a moisturizing agent together with other ingredients such as beeswax, shea butter, olive oil and coconut oil. Honey has anti-bacterial, anti-fungal and natural oxidant properties that are suitable to be added in the formulation of lip balm. Numerous studies have been conducted to investigate the stability of natural lip balm or lipstick (Arifin, Bono et al. 2002, Rajin, Bono et al. 2007, Fernandes, Dario et al. 2013). Although the stability test of lip balm is well understood, no research has been performed for formulation of beeswax and stingless bee honey. Therefore, the objective of this study was to establish the formulation of beeswax and stingless honey based moisturising lip balm.

In this work, natural-ingredient based lip balm was produced. Beeswax, shea butter, coconut oil, olive oil, honey and natural colorant were used to prepare the formulations. The effect of ingredient composition on lip balm's physico-chemical properties such as melting point, stability, moisture content, microbial content and color intensity were studied. The consumers acceptance were also investigated to determine the best formulation that could be produced and sold.

Materials and Methods

Materials

Stingless bee honey was obtained from nursery in Gambang (Kuantan, Malaysia). Beeswax, shea butter, virgin coconut oil, olive oil, fruits flavoring, and preservative were obtained from Soap Lab Malaysia. Strawberry, dragon fruit, and pomegranate were obtained from Giant Supermarket in Kuantan. Ethanol (95% purity) and nutrient agar were purchased from Merck (Darmstadt, Germany).

Fruits juice preparation

Fresh fruits were washed with water to remove impurities such as dust, grit and dirt. Then, fruits were peeled, chopped and weighed. A 100 g of fruits pieces were blended in the laboratory blender until it become a smooth puree. The puree was divided equally among centrifuge bottles and were centrifuged at 10,000 rpm for 10 min or until the pulp has entirely separated from the juice. The juice was filtered to remove remaining pulp and transferred to containers, sealed and stored at -18C until use (Harris 2007).

Betalain extraction

Fresh fruits were washed with water to remove impurities such as dust, grit and dirt. Then, fruits were peeled and about 100g of fruits were weighed and cut into small pieces. These small pieces fruits were added to distillation flask and mixed with 300 ml of 98% ethanol. The mixture was placed in a water bath at 40°C for 4 hrs to leach out betalain. Then, the mixture was filtered to separate the

betalain solution and paste. The solution was distilled and ethanol was collected as condensate and betalain product was left in the flask. The product was dried at 70-80°C for 30 min and weighed (Shune and Khin 2014).

Lip balm formulation

Beeswax and shea butter were heated with an indirect flame (by double boiling) at temperature 75-85 °C with continuous stirring. After the wax melted, virgin coconut oil, olive oil and honey were added into the mixture. Then, the natural colorant from fruit juice or betalain extract was added and heated. Stirring was continued until a proper dispersion was achieved. Finally, the hot mixture was poured into lipstick molds and refrigerated at 5°C for one h and stored at room temperature for 48 hrs to stabilize the lip balm (Alessandrea & Michelli, 2013). The experiment was repeated using different composition of beeswax, shea butter, virgin coconut oil, olive oil, honey and colorant to obtain the stable formulation.

Analysis parameters

Melting point

The melting point of the lip balm was observed with SMPI Melting Point Apparatus Model SMP10. A small quantity of lip balm was placed into the capillary tube of the apparatus, and heated. The melting point was the point where the first liquid formation occurred (Awang et al., 2002).

Stability test

The lip balm formulation was evaluated for 30 days under the conditions of room temperature (25.0 ± 2.0 °C), oven (40 ± 2.0) and refrigerator (5 ± 1.0). The characteristic such as appearance was assessed on the 3rd, 7th, 15th and 30th (Alessandrea & Michelli, 2013).

Moisture analysis for lip balm

The moisture of the lip balm was determined by its moisture content. The moisture loss of lip balm was determined by AOAC method. Lip balm was weighed and recorded as 'wet weight of sample'. Then, the wet sample was dried in an oven (CE Model 30L) at 70°C for 1 h. The sample was left to cool down at room temperature, and was weighed again to record the dry weight of the sample. The moisture content of the sample was quantified using the following formula:

$$W\% = (A-B)/A \times 100\% \quad \text{eq.(1)}$$

Where W% = moisture loss to the surrounding

A = wet weight of sample

B = dry weight of sample

Moisture analysis for lips

Moisture analysis for lips were done by using equipment called Digital Moisture Monitor for Skin (Model SK-IV). Lips of respondents were first ensured to be clean and dry. Then moisture level of respondents' lips were measured before applying the lip balm. Then, lip balm was applied to the lips of the respondents. After ten minutes, moisture of the lips were measured. Moisture content was analyzed using (eq 1).

Colour intensity

For colour intensity test, lip balm formulated from fruit juice and betalain extract were tested to identify which method gives the best colour. Lip balm was placed in a tray of Lovibond Tintometer (Model F Calorimeter) and the intensity of colour range was determined (Norazlin et al, 2015).

Sensory test

The objective of the sensory test was to evaluate the quality of product acceptance by the consumer. The consumer acceptance was investigated using the Ranking Test. The sensory test was conducted using lip balm with different composition of beeswax, shea butter, oil and honey. Sensory test was carried out using 50 participants. The respondents were asked to rank the acceptance from 1 to 3; 1-bad, 2-average and 3-good based on the hardness, spreadability and moisture of the lip balms (Awang et al., 2002). Hedonic scale is used to check the overall consumer acceptance while ranking test is used to check the best formulation.

Microbial test

Preparation of nutrient agar and agar plates

Nutrient agar powder (23 g) was weighed and mixed with 1 L of distilled water in 1L Schott Bottle. The mixture was autoclaved at 121°C for 20 min. For the preparation of agar plates, 15-20 ml of warm sterile nutrient agar was poured into the petri dishes and left to solidify at room temperature.

Spread plate technique

Firsly, work place and apparatus were sprayed with ethanol to decrease the contamination. Lip balm was scoop out from the mould and placed at the center of agar plate. The lip balm was spreaded evenly on the surface of the agar by using sterile plastic spreader (hockey stick). After lip balm was fully spread on the surface of agar, petri plate was incubated in an inverted position at 30°C for 24 hrs(Diane 2011).

Results and Discussion

Melting point

Melting point of the lip balm was affected by the composition of beeswax in the formulation. Three types of lip balm with beeswax composition of 1.25, 2.50 and

3.25 gram gave different results. As the beeswax composition increased the melting point of the lip balm also increased (Figure 1). This finding is in agreement with Mariani et al (2007) that the beeswax enhance the melting point of the lip balm.

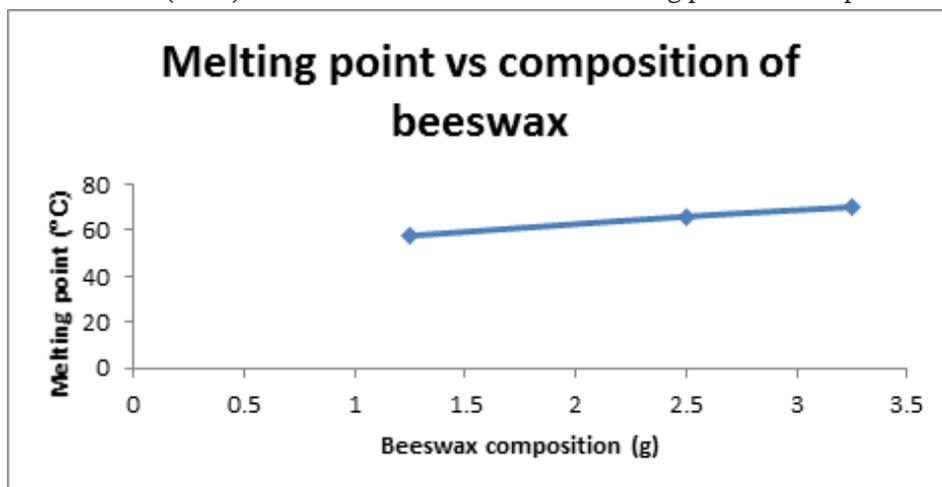


Figure 1. Effect of beeswax composition on the melting point of the lip balm

The melting point of the lip balm of this study was between 58 to 70°C. According to Mariani et al. (2007) melting point of lip balm is generally within the ranges of 55 to 75°C. Thus, the lip balms of this study fall within the general melting point ranges of lip balms. Therefore, lip balms with higher composition of beeswax increased in hardness which resulted in higher melting point.

Stability

Six types of lip balm with same formulation were placed at different conditions to study the stability. Two lip balms were placed at room temperature, two lip balms were placed at oven and two lip balms were placed at refrigerator. Result of stability test included appearance, color, odor and spreadability of the lip balm (Table 1). The appearance of lip balm was marked as Normal and Modified. In Normal condition, there were no change of appearance and in Modified condition, the appearance changed on the surface of the lip balm. The appearance was normal under refrigerator and room temperature conditions, but modified under oven condition. Blooming and streaking was not observed in the appearance of the lip balm stored at all conditions. In Oven Condition, lip balm appeared sweat and the texture was soft due to effect of high temperature. These changes were classified as Modified in Table 1.

As for color of the lip balm in three conditions, no color change was observed at room temperature and refrigerated condition. However, color of the lip balm changed at oven condition. Meanwhile for the odor, strawberry and raspberry flavoring added to the lip balm remained stable throughout 30 days of testing under all temperature conditions.

Lastly for spreadability test, spreadability of lip balm was assumed good for formulations stored under refrigerator and room temperature. Lip balm showed

uniformity and perfect application without fragmenting or deformation under these conditions. On the other hand, under oven condition, spreadability of lip balm was considered bad since the application was not uniform, and fragments and deformation of the lip balm was observed during test.

Table 1
Evaluation of physical characteristics and spreadability of lip balm in stability test

Parameters	Storage conditions											
	Refrigerator				Room Temperature				Oven			
	3d	7d	15d	30d	3d	7d	15d	30d	3d	7d	15d	30d
Appearance	N	N	N	N	N	N	N	N	M	M	M	M
Color	N	N	N	N	N	N	N	N	M	M	M	M
Odor	N	N	N	N	N	N	N	N	N	N	N	N
Spreadability	G	G	G	G	G	G	G	G	B	B	B	B

Conditions: Refrigerator (5.0 ± 1.0 °C), Room temperature (25.0 ± 2.0 °C), Oven (40 ± 2.0 °C)

Appearance, Color, Odor: N-Normal, M-Modified

Spreadability: G-Good, B-Bad

In summary, appearance, color, odor and spreadability of the lip balm were found normal and good at room temperature and refrigerated condition (Table 1). The lip balm must not be stored at oven condition (40 ± 2.0 °C).

Moisture

Moisture analysis for lip balm

The main point of honey lip balm which promotes moist property was determined by moisture content. Four types of lip balm with honey composition of 1.0, 1.5, 2.0 and 2.5 gram were tested. The result of moisture loss to the surrounding was shown in Figure 2.

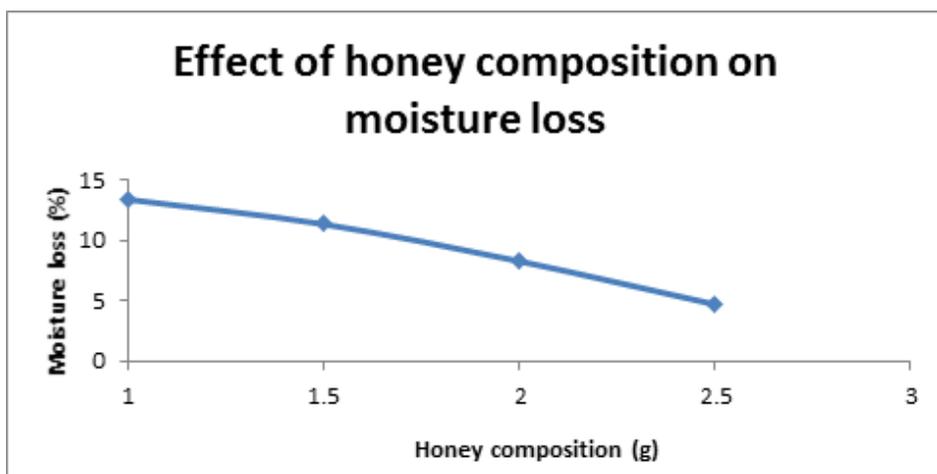


Figure 2. Effect of honey composition to the moisture loss of lip balm

Figure 2 shows that as honey composition increases, the moisture loss of lip balm decreases. This is because as composition of honey increases, the moisture of the lip balm also increases. Honey composition consists of sugars, lactic acid and amino acids that behave as humectants. Therefore, honey enhanced humectant properties in the formulation. Besides that, humectant activity is also due to presence of small polyol molecules such as glycerin, propylene glycol and sorbitol (Isla et al, 2013).

Moisture analysis for lips

Since function of lip balm is to moisturize the lips, it is essential to measure the lips moisture after applying the lip balm. Moisture content of the lips of 15 respondents were measured before and after the application of lip balm. The results were shown in Figure 3.

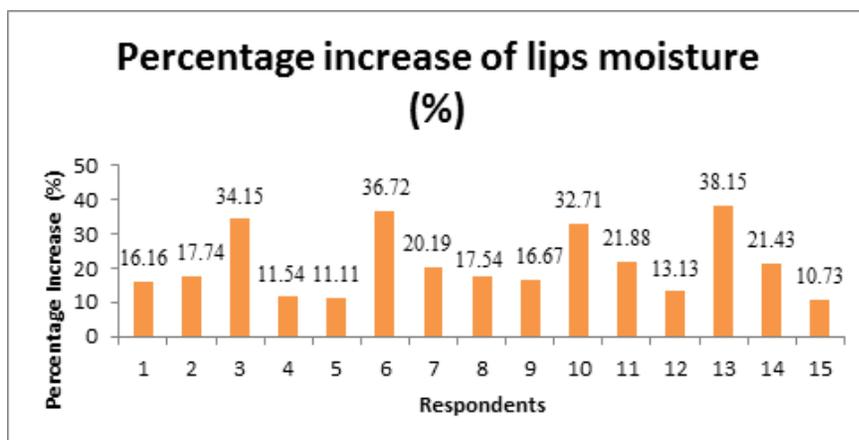


Figure 3. Percentage increase of lips moisture

For the study of lips moisture, the result obtained had achieved the objective of this study which is formulating a moisturizing lip balm. All the lips moisture of respondents tested showed the different level of lips moisture before and after applying lip balm, the moisture level increased between the ranges of 10.73% to 38.15%. The results indicate that there was difference in lips moisture of people who did not use lip balm and who use lip balm. Honey has hygroscopic properties, which mean it can absorb moisture in the air when being exposed (Manisha and Shyamapada 2011).

Sensory test for consumer acceptance

The objective of sensory test was to investigate the consumer acceptability towards lip balm samples. Table 2 shows the best three formulations of lip balm selected by respondents in the Ranking Test.

Table 2. Result of the Ranking Test; S1 indicates the formulation of Sample 1, S2 indicates the formulation of Sample 2 and S3 indicates the formulation of Sample 3.

Table 2
Evaluation of physical characteristics and spreadability of lip balm in stability test

Samples/	S1		S2		S3	
	g	%	g	%	g	%
Beeswax	2.5	26.32	1.25	17.24	2.5	28.57
Shea butter	2.5	26.32	2.5	34.48	1.25	14.29
Oil	2.5	26.32	2.5	34.48	2.5	28.57
Colorant	2.0	21.05	1.0	13.79	2.5	28.57
Total	9.5	100.00	7.25	100.00	8.75	100

After the best formulations of lip balm were achieved, the Hedonic Scale test was used to research the overall consumer acceptance for the best formulation. Participants were asked to scale the samples based on hardness, moisture and spreadability. The scale started from bad (1), average (2) and good (3). Figure 4 showed the result of samples that got good marks for the three attributions of hardness, moisture and spreadability.

From the result of sensory test, respondents preferred (70%) the hardness in S1 formulation compared to S2 and S3 formulations. In terms of moisture, respondents (80%) selected S2 formulation for the lip balm. For spreadability, 80% and 70% respondents accepted S1 and S2 formulation, respectively. S1 formulation was found to give easy and uniform spread when applied on lips. According to overall acceptance, the most preferred formulation was S1 which obtained two highest score compared to S2 and S3. Thus, S1 and S2 formulation both could be accepted by consumers as they had 40-80% preference by respondents in terms of hardness, moisture and spreadability. S3 formulation was rejected due to poor acceptance; only 20-30% participants liked its hardness, moisture and spreadability.

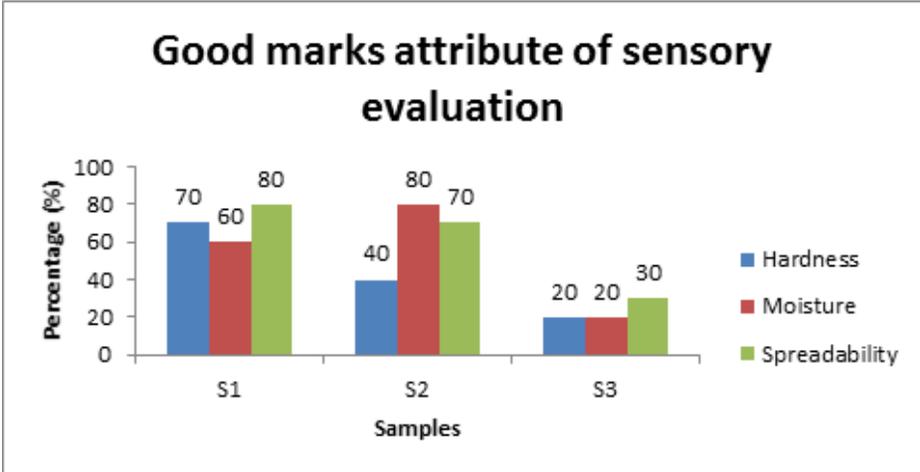


Figure 4. Good marks attribute of sensory test

Color intensity

For color intensity, lip balms had different color intensity depending on which colorants were used, whether from fruit juice, extract of betalain and color powder. The results of color intensity of lip balm were showed in Figure 5-7.



Figure 5. Lip balm by using dragon fruit

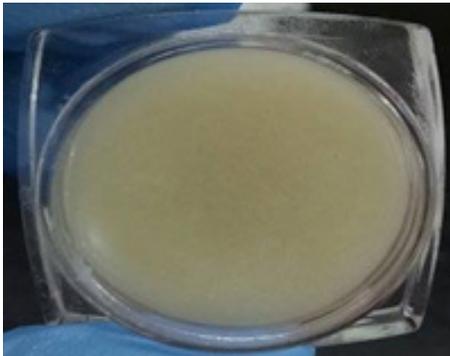


Figure 6. Lip balm by using betalain extract and strawberry juice

A-natural colorant from dragon fruit, B-natural colorant from strawberry and C-powder colorant



Figure 7. Color comparison between

From Figure 5, dragon fruit lip balm gives color of light purple while strawberry lip balm give color of pale pink. Meanwhile for Figure 6, lip balm of betalain extract gives color of light yellow. Although the extraction of betalain from dragon fruit produced red liquid when mixed with lip balm formulation, the liquid immersed with other main ingredients like beeswax and shea butter and did not give red color effect to the lip balm. Lastly, for Figure 7, it shows the comparison between natural colorant and powder colorant lip balms. Powder colorant gives good intensity of color compared to natural colorant from fruits.

Microbial test

In microbial test compared the effectiveness of preservatives added in the lip balm to prevent growth of microorganism. Spread plate technique was conducted to detect the presence of microbes in the lip balm. Figure 8 showed the results of lip balm with and without added preservatives to the lip balm.

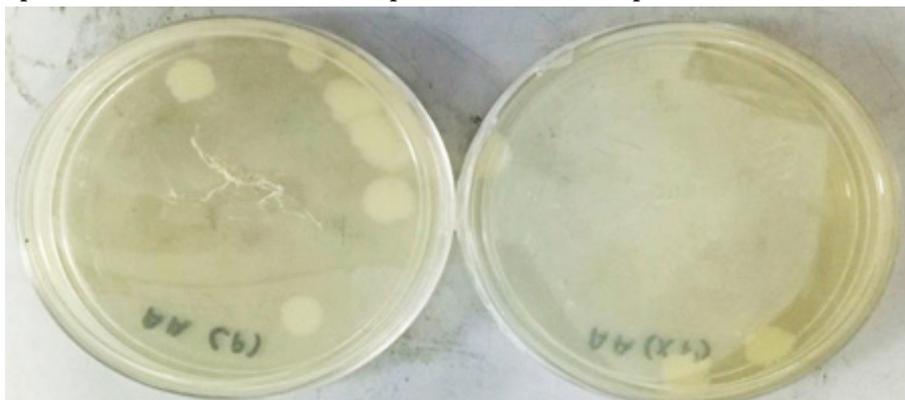


Figure 8. Result of spread plate technique

The result showed that lip balm formulation that contained preservative had low microbial count compared to lip balm formulation that do not contain preservative. Low microbial count in spite of addition of preservative is because the lip balm was not a sterile cosmetic and was not produced by aseptic processes and thus is not expected to be totally free from contamination of microbes (Nicole et al, 2014).

Other Measurement Tables

A1-Data of melting point

Beeswax composition(gram)	Melting point (°C)
1.25	58
2.5	66
3.25	70

A2- Data of moisture content for lip balm

Composition of honey (gram)	Moisture loss (W %)
1.0	13.40
1.5	11.34
2.0	8.25
2.5	4.64

A3- Data of moisture content for lips

Respondents	Lips moisture before applying lip balm	Lips moisture after applying lip balm	Percentage increase (%)
1	30.6	36.5	16.16
2	26.5	31.2	17.74
3	16.4	22	34.15
4	31.2	34.8	11.54
5	25.2	28.0	11.11
6	30.5	41.7	36.72
7	31.2	37.5	20.19
8	28.5	33.5	17.54
9	31.2	36.4	16.67
10	26.9	35.7	32.71
11	25.6	31.2	21.88
12	29.7	33.6	13.13
13	27	37.3	38.15
14	23.8	28.9	21.43
15	20.5	22.7	10.73

A4- Data of sensory test for Sample 1

Attribute	Percentage (%)		
	Bad	Average	Good
Hardness	•	۳•	۷•
Moisture	•	۴•	۶•
Spreadability	•	۲•	۸•

A5- Data of sensory test for Sample 2

Attribute	Percentage (%)		
	Bad	Average	Good
Hardness	10	50	40
Moisture	0	20	80
Spreadability	10	20	70

A6- Data of sensory test for Sample 3

Attribute	Percentage (%)		
	Bad	Average	Good
Hardness	20	60	20
Moisture	20	80	20
Spreadability	30	40	30

Conclusion

From the study, the formulation by using natural based ingredients was considered success and achieved the target. The formulation of lip balm from beeswax, shea butter and oil with ratio 1:1:1 gives the best physico-chemical properties and has been accepted by the consumer.

The formulation of lip balm from beeswax, shea butter and oil with ratio of 1:1:1 has melting point of 66 °C and has stable condition at low and room temperature condition. The presence of honey in the formulation enhanced the moisture content of the lip balm. The higher the honey composition, the higher the moisture content of lip balm. Besides that, for color intensity, natural colorant from fruit juice and extraction give the color to the lip balm but the color intensity was lower compared to powder colorant. Lastly, the formulation of lip balm from beeswax, shea butter and oil with ratio 1:1:1 was most preferred formulation by participants. Thus most likely formulation that will be accepted by the consumers when the product is marketed is the lip balm formulation containing 1:1:1 ratio of beeswax, shea butter and oil.

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