Characterization of solid perfume based on Cocoa Butter with Jasmine Oil as fragrance

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Abstract. Cocoa butter can be obtained from by-product of chocolate powder processing from the result of mechanical pressing using hydraulic press. As a source of vitamin E, cocoa butter in cosmetic has some advantages which are to soften and moisturize the skin. Meanwhile, jasmine can be used as a fragrance in perfume and cosmetics. In this study, cocoa butter was used as raw material for solid perfume. Its optimal concentration in the product was determined. Solid perfume was made by melting cocoa butter and beeswax at 90°C followed by adding jasmine oil as fragrance. The formulation variation of solid perfume was done with cocoa butter concentration 10%, 20%, 30%, 40% and 50% (w/w) and addition of patchouli oil as fragrance fixative agent. The following tests were carried out on the products: functional group analysis, hardness test, homogeneity test, physical stability test, melting point test, antioxidant test, and organoleptic test. The result showed that the variation of cocoa butter concentration affected the physical characteristics of the solid perfume. The optimum product of solid perfume based on panelists’ organoleptic test was achieved with cocoa butter concentration of 30%. It had an organoleptic score of 3.63, antioxidant activity with IC50 value of 201.98μg/mL, hardness penetration 14.16 mm/second, melting point 90°C and pH value 4.

1. INTRODUCTION
Cocoa butter, known as theobroma oil, is a natural fat obtained from cocoa beans processing by mechanical pressing using a hydraulic press or by chemical processing such as extraction. Cocoa butter is widely used as basic ingredient in food, pharmaceutical and cosmetic industries. It contains compounds that are beneficial for skin, namely stearic acid, palmitic acid, oleic acid also vitamins A, C and E. The addition of cocoa butter in cosmetics can moisturize, smoothen, and soften the skin. Cocoa butter has melting point around 30-35°C which is lower than the temperature of the human body thus it seeps into the skin quickly [1]. Cacao butter consists of fatty acid mostly oleic acid, triacylglycerols and γ-tocopherol [2]. Oleic acid can disrupt skin barriers thus it can enhance permeability on stratum corneum layer [3]. The fat content prevents evaporation on the skin and provides lubrication for skin surface which maintains the lipid content.
of the skin thus it has a good texture. Cocoa butter also has similar lipid compositions with skin lipid composition [4].

Many cosmetic products use cocoa butter as raw material such as cream, sunscreen, lipstick, soap and shampoo [5]. In this study, cocoa butter was used as raw material of solid perfume. Solid perfume was made by mixing the wax base, carrier oil, and essential oil as fragrances followed heating process. The liquid product was poured into a container and allowed to harden at room temperature. Hardiyati et al., has made solid perfume using the essential oils aroma of vanilla, jasmine and sweet orange. The ingredients of solid perfume consisted of carrageenan, stearic acid and cetyl alcohol as wax base, triethanolamine, acrylonitrile butadiene styrene (ABS), preservatives and patchouli essential oils as fixatives [6].

Essential oils are widely used for aromatherapy through respiration which allows to penetrate into the human body. Cosmetics with aromatherapy purpose used essential oils in skin, body, face and hair products. It is also safe to use as raw material for perfume [7], [8]. One of the essential oils that often used as fragrances or perfume is jasmine oil. Besides the nice aroma, jasmine oil also has a pharmacological function, namely increase breathing speed, increase oxygen absorption in the blood, reduce depression and improve human mood [9].

Studies related to solid perfume made from cocoa butter are still rarely found. Therefore, in this study, optimization of solid perfume formulation was carried out using cocoa butter as raw material and jasmine oil as fragrance. The solid form aims for practical use and storage as well as increase its stability. In this study, characterization of cacao butter as raw material was conducted by antioxidant test, saponification numbers and acid numbers test and analysis of the chemical components of jasmine oil essential oils. Characterization carried out on solid perfume products included functional group analysis, pH test, hardness level test, homogeneity test, stability test and organoleptic test or hedonic test.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in this study namely, beeswax technical grade (brataco, Indonesia), jasmine oil (brataco, indonesia), avocado oil (brataco, Indonesia), patchouli oil from Chemical Research Center LIPI, Indonesia and cocoa butter from Biotechnology Research Center LIPI, Indonesia.

2.2. Methods

2.2.1. The Process of Making Solid Perfume. Cocoa butter and beeswax were melted at 90oC. Jasmine oil as fragrance was mixed with avocado oil and then added to wax compound. After that, patchouli oil was added gradually. The mixture was stirred until homogeneous for 30 minutes then cooled and allowed to harden at room temperature. The composition of solid perfume is shown in Table 1.

| Table 1. Variations in Solid Perfume Composition (% weight). |
|-----------------|---|---|---|---|---|---|---|---|---|
| Materials       | F1 | F2 | F3 | F4 | F5 | F6 | F7 | F8 | F9 |
| Cocoa butter    | 10 | 20 | 30 | 40 | 50 | 10 | 20 | 30 | 40 |
| Beeswax         | 50 | 40 | 30 | 20 | 10 | 50 | 40 | 30 | 20 |
| Avocado Oil     | 10 | 10 | 10 | 10 | 10 | 9.9| 9.9| 9.9| 9.9|
| Jasmine Oil     | 30 | 30 | 30 | 30 | 30 | 30 | 30 | 30 | 30 |
| Patchouli Oil   | -  | -  | -  | -  | -  | 0.1| 0.1| 0.1| 0.1|
2.2.2. Characterization. Cocoa butter as raw material was tested for acid number based on SNI 3748:2009, saponification number based on SNI 3748:2009 and antioxidant test using 2,2-diphenyl-1-picrylhydrazyl (DPPH) method. Jasmine essential oil and patchouli oil were analyzed for the chemical compounds by Gas Chromatography-Mass Spectrometry (GC-MS) Agilent 7890B with MSD 5977A and refractive index by refractometer Atago RX-5000α. Solid perfume products were tested for their physical properties that were pH test using universal pH indicator, hardness level using penetrometer, melting point using the capillary pipe method and analysis of chemical functional groups using Fourier Transform Infra-Red (FTIR) Shimadzu IR Prestige-21.

3. RESULTS AND DISCUSSION

3.1. Acid Number and Saponification Number of Cocoa butter
Characterization of acid number and saponification number of cocoa butter raw material was carried out to determine the quality of the material compared with the SNI 3748:2009 quality standard. Characterization of acid number and saponification number of cocoa butter are shown in Table 2.

| Parameter                        | Result    | SNI Standard |
|----------------------------------|-----------|--------------|
| Acid Number (%)                  | 1.19      | Max 1.75     |
| Saponification Number (mg/KOH/gr fat) | 192.28   | 181-198      |

Based on the results, cocoa butter used in this study had good quality with acid and saponification number below the SNI standard’ threshold. The presence of excess free fatty acids in cocoa products were undesirable because it indicated low quality product due to moist and unclean storage [10]. The saponification number indicates the amount of fatty acids. Cocoa butter with saponification fat ranging from 190-200 mgKOH/gr contained fatty acids with moderate molecular weight [11].

3.2. Analysis of Essential Oils’ Chemical Compounds
Essential oils used in this study were jasmine oil as fragrance and patchouli oil as fixative or scent binder. Samples were analyzed using GCMS to determine the content of chemical compounds in each essential oil. Chromatogram images along with details of jasmine oil compounds are shown in Figure 1 and Table 3.

Based on Edris et al., study, there were about 47 compound components from the extraction of Jasmine Jasminum Sambac flowers from Egypt. The dominant components included benzyl acetate 14.2%; indole 13.4%; E-E-a-farnesene 13.1%; Z-3-hexenyl benzoate 9.4%; benzyl alcohol 8.4%; and linalool 6.3%. Hidayat et al., found 38 components from Jasminum Sambac extraction and the dominant components were benzyl acetate 15.78%; linalil acetate 10.32%; cis jasmone 10.04%; Z-jasmone 8.32%; and linalool 6.10%. The composition of chemical compounds in the same plant can be differ depends on the level of impurity, plant characteristic and plant origin [12], [13].

In this study, commercial jasmine oil was used. There were several similar compounds with the previous study such as linalool, benzyl acetate and benzyl alcohol. The dominant compound was amylcinnamaldehyde, which was a derivative of cinnamic acid. Various derivatives of cinnamic acid were used as treatment material for skin, hair, antimicrobial ingredients and widely used as raw materials for perfumes. Amylcinnamaldehyde compound characterized as a pale yellow liquid with a floral aroma that gave the impression of jasmine aroma in cosmetic products [14].
Figure 1. GC-MS Chromatogram of Jasmine Oil

Table 3. Chemical Compounds of Jasmine Oil

| Retention Time | % Area | Compound Name                                      |
|----------------|--------|---------------------------------------------------|
| 7.148          | 14.43  | 2-propanol, 1,1'-oxybis-                           |
| 7.229          | 2.72   | Benzyl alcohol                                    |
| 7.438          | 11.92  | 1-propanol, 2-(2-hydroxypropoxy)-                 |
| 8.239          | 6.15   | Linalool                                          |
| 8.489          | 9.99   | Phenylethyl alcohol                               |
| 9.237          | 18.88  | Benzyl acetate                                    |
| 12.883         | 1.84   | Benzethanol, alpha, 4-dimethyl                    |
| 15.542         | 18.97  | Cinnamaldehyde, alpha-pentyl                      |
| 17.888         | 3.13   | Benzene, 1-(1,1-dimethylethyl)-3,5-dimethyl-2,4,6-trinitro- |

The results of the patchouli oil chemical compounds characterization are shown in Figure 2 and Table 4. The dominant compositions based on characterization were alpha.-Guaiene, delta.-Guaiene and Patchouli alcohol. The resulting patchouli oil composition characterization was similar to Ermaya et al (2019) where the patchouli oil content consisted of Patchouli alcohol 42.75%, Delta-Guaiene 28.3%, Azulene 20.48%, Trans Caryophellene 11.84%, Seychellence 10.77%, Nephtalene 8.02%, Cycloheptane 6.03 % and Caryophyllene 5.73% [15].

Table 4. Chemical Compounds in Patchouli Oil

| Retention Time | % Area | Compound Name       |
|----------------|--------|---------------------|
| 12.359         | 3.16   | Beta-Patchoulene    |
| 12.862         | 3.05   | Caryophyllene       |
| 13.090         | 14.31  | alpha.-Guaiene      |
| 13.191         | 5.69   | Seychellene         |
| 13.354         | 7.24   | Alpha-Patchoulene   |
| 13.947         | 21.78  | delta.-Guaiene      |
| 15.849         | 32.57  | Patchoul alcohol    |
| 16.344         | 3.32   | Pogostone           |
3.3. Essential Oil Refractive Index
Refractive index is one of the parameters of quality analysis of essential oil based on SNI 06-2385-2006. Refractive index of Jasmine oil was about 1.47028-1.47038. This value was similar to Ginting 2019 with refractive index of Jasmine oil 1.478 whereas in general the value of Jasmine oil refractive index was about 1.470-1.492 [16].

3.4. Chemical Functional Group of Solid Perfume
The functional group of solid perfume and the raw materials were analyzed using FTIR and the results of the analysis are shown in Figure 3. Based on the analysis results, there were absorbance at 3377 cm⁻¹ on solid perfumes that corresponded to the -OH functional groups. The cocoa butter showed the wavenumber at 3610 cm⁻¹, jasmine oil at 3369 cm⁻¹ and patchouli oil at 3606.89 cm⁻¹ where these absorptions indicated the -OH group. Other peaks occurred that showed -CH₃ and -CH₂ groups in solid perfume at wavenumbers 2862 cm⁻¹ and 2927 cm⁻¹, cocoa butter at 2860.43 cm⁻¹ and 2927.94 cm⁻¹, jasmine oil at 2881.65 cm⁻¹ and 2953 cm⁻¹, and patchouli oil at 2941 cm⁻¹. The presence of wavenumber in solid perfume at 1739 cm⁻¹, cocoa butter at 1735.93 cm⁻¹, jasmine oil at 1680 cm⁻¹, and oil at 1732 cm⁻¹ corresponded to the content of -CO aldehyde group in each sample.

Based on the results of FTIR analysis, solid perfume was produced through the physical mixing process of various raw materials, of which there were no new functional groups that indicated the formation of new compound. The functional groups found in raw materials that were cocoa butter, jasmine oil and patchouli oil were also found in solid perfume product.
3.5. pH Test of Solid Perfume Formulation

pH standard for topical preparation in contact with skin was about 4-8 [17]. The pH value was expected not to be too acidic because it can cause irritation and not too alkaline because it can cause scaly skin. The resulting solid perfume was tested using universal pH paper and had pH value of 4. This pH value was considered safe for topical preparation for human skin application. Moreover, the content of this solid perfume was cocoa butter which contained fatty acids that similar to fatty acids composition on the skin [1].

3.6. Homogeneity of Solid Perfume Formulations

Homogeneity test of solid perfume was conducted by applying the samples to a flat glass surface as shown in Figure 4. The results of this test showed there were no coarse grains on the surface of the glass. According to Nurany et al (2018) homogeneous product had no coarse grain on the glass surface that had been smeared by the product [18].

3.7. Hardness Level of Solid Perfume Formulation

The effect of concentration variations of cocoa butter on solid perfume products hardness level can be seen in Figure 5.
As shown in Figure 5, the penetration value would be higher when more cocoa butter was added. The highest penetration rate was at 50% cocoa butter concentration by value 13.23-14.16 mm/sec and the lowest was at 10% cocoa butter concentration by value 0.46-0.66 mm/sec. The addition of cocoa butter made the solid perfume product soften. The presence of cocoa butter in formulation was an important factor to adjust the texture and melting point. The cocoa butter affected the composition of solid perfume crystals into a more stable polymorphic arrangement. More amount of cocoa butter increased the consistency and density properties [19]. Cocoa butter can filled the space between the mixed ingredients which caused lubrication process that lead to decrease of rheological properties, texture and thermal properties [20].

3.8. Melting Point of Solid Perfume Formulation
The melting point is one of quality testing for solid perfume products. This test represents the stability of product quality during manufacture, storage, and usage processes [21]. Melting point results for all solid perfume variations are shown in Table 5.

**Table 5. Melting Point for Solid Perfume Products.**

| Sample | Melting Point (°C) |
|--------|-------------------|
| F1     | 100               |
| F2     | 100               |
| F3     | 90                |
| F4     | 80                |
| F5     | 74                |
| F6     | 115               |
| F7     | 100               |
| F8     | 100               |
| F9     | 80                |
| F10    | 85                |
Melting point from solid perfume products generally ranged above 70°C. Solid perfume with the highest melting point was the formulation with the lowest cocoa butter concentration by value 10%. The increased of melting point proportional with the increased of product hardness [22]. This corresponded to the decreased of rheological properties along with increased cacao butter concentration. It caused a decrease in the solid perfume products’ melting point at high cocoa butter concentration.

3.9. Antioxidant Activity Test of Solid Perfume
Cocoa butter and solid perfume product were tested for antioxidants by DPPH method using UV-Vis Spectrophotometer with a maximum wavelength of 515 nm and used vitamin C as positive control. The results of antioxidant activity tests are shown in Table 6. IC₅₀ value in cocoa butter was 168 µg/mL and in solid perfume was 201.98 µg/mL, this value was categorized as weak antioxidant. Based on Molyneux (2004), very strong antioxidants were those that have IC₅₀ values less than 50 µg/mL, strong antioxidants with IC₅₀ 50-100 µg/ml, medium antioxidants with IC₅₀ 101-150 µg/ml and low antioxidants with IC₅₀ 151-200 µg/mL [23]. Pure cocoa products had high antioxidant value due to the presence of polyphenol content, while cocoa butter was a by-product of the cocoa bean process, thus the antioxidant activity of cocoa butter was lower than pure cocoa [24].

| Sample                  | IC₅₀ (µg/ml) |
|-------------------------|-------------|
| Cocoa butter            | 168         |
| Solid Perfume           | 201.98      |
| Vitamin C (positive control) | 44.6       |

3.10. Stability Test of Solid Perfume Product
The stability test aimed to evaluate the product characteristics by observing the physical endurance of the product under certain condition. The test on solid perfume was carried out at room temperature by observing changes in texture, color and aroma for 4 weeks. The results of solid perfume products stability are shown in Table 7.

| Solid Perfume | Weeks | Texture                        | Color                               | Aroma                                      |
|---------------|-------|--------------------------------|-------------------------------------|---------------------------------------------|
| F1            | 1-4   | Solid, hard, and difficult to apply | Yellowish white, stable until the fourth week | Typical jasmine, stable until the fourth week |
| F2            | 1-4   | Solid, hard, and difficult to apply | Yellowish white, stable until the fourth week | Typical jasmine, stable until the fourth week |
| F3            | 1-4   | Solid, a little soft and easy to apply | Yellowish white, stable until the fourth week | Typical jasmine, stable until the fourth week |
| F4            | 1-4   | Creamy, a little soft and uncomfortable to apply | Yellowish white, stable until the fourth week | Typical jasmine, stable until the fourth week |
| F5            | 1-4   | Creamy, soft and uncomfortable to apply | Yellowish white, stable until the fourth week | Typical jasmine, stable until the fourth week |
| F6            | 1-4   | Solid, hard, and difficult to apply | Yellowish white, stable until the fourth week | Typical jasmine and a little patchouli aroma, stable until the fourth week |
| F7            | 1-4   | Solid, hard, and difficult to apply | Yellowish white, stable until the fourth week | Typical jasmine and a little patchouli aroma, stable until the fourth week |
9

Overall, the observation for 4 weeks on all formulations had good stability for color and aroma parameters, but had a different texture consistency. The sensory of preparations with high cocoa butter concentration had soft texture which was uncomfortable when it applied to the skin. Product with high beeswax concentration had hard texture which was difficult to apply. The addition of waxes such as beeswax and cocoa butter greatly affected the consistency of the final product [22]. The addition of patchouli oil gave its distinctive aroma and intensified the scent of jasmine oil. It because the function of patchouli oil was to hold other scents thus the fragrance didn’t disappear quickly and long last [23].

3.11. Organoleptic Test of Solid Perfume Products

Organoleptic test was carried out on 10 formulations. Organoleptic test refers to SNI 01-2346-2006 about organoleptic and/or sensory testing. Organoleptic test was a method of testing using the five human senses. This test involved 30 untrained panelists and they were given an assessment with the parameters written on the questionnaire. The test was done by selecting the assessment score with range 1-5 where each number indicated the level of preference for the tested solid perfume product. Organoleptic test results are shown in Table 8.

Table 8. Organoleptic Test of Solid Perfume.

| Sample | Texture | Color | Aroma | Appearance | Moisture | Comfort | Homogeneity | General |
|--------|---------|-------|-------|------------|----------|---------|-------------|---------|
| F1     | 2.80    | 3.37  | 3.23  | 2.87       | 2.40     | 2.63    | 3.40        | 2.87    |
| F2     | 3.07    | 3.50  | 3.50  | 3.40       | 2.77     | 3.17    | 3.97        | 3.03    |
| F3     | 3.60    | 3.73  | 3.33  | 3.67       | 3.47     | 3.67    | 4.03        | 3.63    |
| F4     | 3.23    | 3.13  | 2.93  | 3.03       | 3.17     | 3.00    | 3.40        | 2.90    |
| F5     | 1.80    | 2.03  | 1.87  | 1.57       | 2.00     | 1.83    | 2.17        | 1.50    |
| F6     | 2.70    | 3.37  | 3.67  | 2.87       | 2.23     | 2.67    | 3.83        | 2.87    |
| F7     | 3.00    | 3.63  | 3.47  | 3.27       | 2.93     | 3.07    | 3.73        | 3.20    |
| F8     | 3.60    | 3.80  | 3.70  | 3.70       | 3.70     | 3.67    | 4.10        | 3.37    |
| F9     | 3.47    | 3.20  | 3.03  | 3.37       | 3.47     | 3.30    | 3.80        | 3.30    |
| F10    | 1.50    | 1.60  | 2.07  | 1.67       | 1.73     | 1.83    | 2.43        | 1.67    |

The most preferable solid perfume formula based on organoleptic test was formula F3, with cocoa butter concentration 30% without the addition of patchouli oil by preference value 3.63. Panelists did not like solid perfume with cocoa butter concentration 50% which had the lowest preference value of 1.50.

Based on statistical test using one-way ANOVA, there was a significant difference with probability value of 0.000 (P <0.005) which showed that the addition of cocoa butter concentration in solid perfume affected the level of panelist preference. This caused by consistency that was very dependent on the ratio of beeswax and cocoa butter. Organoleptic test process involved a sensory process and also a cognitive process. This
sensory analysis can identify the level of acceptance of a product which was the basis for product development and a reference to the concentration limits of the used ingredients [25].

4. CONCLUSION
The concentration of cocoa butter significantly affected the characteristics of the final product. The process only involved the physical process because there were no new chemical groups formed in the final product. The most preferred formulation based on organoleptic test was product with 30% cocoa butter concentration with a preference level score of 3.63. Solid perfume with 30% cocoa butter concentration had characteristics of penetration level of 14.16 mm/sec, melting point 90°C, pH value of 4 and IC50 201.98 μg/mL. Solid perfume products showed homogeneity and physical stability during 4 weeks observation at room temperature. The optimum formulation of this solid perfume was expected to provide new form aside of liquid conventional perfumes.

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