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Development of Emulsion Gel Sunscreen Containing Olive Oil and Clove Oil

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Abstract.
Sunlight contributes to the healing of multiple health conditions, including in respiratory diseases such as Covid-19, by boosting the immune system. Nevertheless, too much sun exposure can cause sunburn and lead to skin cancer. To minimize the harms of excessive sun exposure, the skin needs to be strictly shielded with the use of sunscreen. This study aimed to develop an olive oil and clove oil emulsion gel preparation and determine its sun protective factor (SPF) value. Olive oil was formulated into an emulsion gel with varying concentrations of 2%, 4%, and 6%, and then 5% of clove oil was added. Physicochemical properties such as organoleptic, homogeneity, pH values, viscosity, and spreadability were tested. Further, UV protection was examined based on determining the SPF value, and the stability of preparation was evaluated using real-time and freeze-thaw methods. The results showed that the preparation had a good physicochemicality, while the SPF value was 20.91 ± 0.29, 22.52 ± 0.52, and 23.39 ± 0.45, respectively. However, the results of the stability test showed a significant change in the pH and SPF value after storage. It can be concluded that the emulsion gel of olive oil and clove oil preparation could be considered to have a medium level of protection of sunscreen.

Keywords: olive oil, clove oil, emulsion gel, sunscreen, UV protection, stability

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1. INTRODUCTION

Sunlight contributes to the healing of multiple health condition including respiratory diseases. Sunlight can maintain the health condition of Covid-19 patients by triggers vitamin D production, which boosting the immune system then slows down the development of the virus in the human body. The higher duration of sunlight exposure was related to more cases of recovery from Covid-19 among patients [1]. This information is affecting the lifestyle behaviors at the global level, including doing sunbathe [2, 3].

But, too much of sunlight may cause harm through its UV, IR, etc. Sunburn (erythema) is the most obvious acute clinical effect and skin cancer is the most important chronic effect [4]. Skin cancer is the most commonly diagnosed cancer in many countries. The use of sunscreen is one of the protections to protect the skin from UV radiation.

Sunscreens protect against the skin-damaging effect of UV radiation through either chemical or physical ingredients, which acts to block UV radiation [5]. Sunscreen can absorb about 85% of sunlight exposure at a wavelength of 290-320nm (UVB rays) [6].

Olive oil and clove oil are essential oils which extracted from plants and have an activity as UV protection. Olive oil has an SPF value of 7.549 [7] and clove oil has an SPF value of 2.4 [8]. Clove oil contain about 73.5 – 96.9% eugenol and olive oil contain about 10 – 20% of tocopherol which can inhibits ROS and RNS formations to protect the skin as antioxidant [9, 10]. These oils can be developed in the preparation of sunscreen formulation as they are moisturizing, scenting, less irritating and less expensive [8].

Emulsion gel is a composite structure consisting of oil droplets within a gel matrix [11]. This system has excellent solubilizing properties for lipophilic and hydrophilic active ingredients and great end-user acceptability due to the pleasant skin sensory. Emulsion gel is vehicle that presents low permeation, keeping the substance active at the skin surface [12]. The aim of this study was the development, characterization and determination of UV protection activity of olive oil and clove oil emulsion gel preparation.

2. MATERIALS AND METHODS

2.1. Materials

Clove oil was purchased from PT. Nusaroma Essential Indonesia and Olive oil was obtained from PT. Lansida, Yogyakarta. Other materials including carbomer, liquid paraffin, triethanolamine (TEA), Tween 80, Span 80, methyl paraben, propyl paraben, propylene glycol, and distilled water in technical grade for emulsion gel preparation. The ethanol pro analysis was used for the SPF activity test.

2.2. Methods

2.2.1. Emulsion Gel Preparation

The oil-in-water emulsion gel was prepared with the composition as shown in Table 1. The carbomer was sprinkled into distilled water, gradually, and developed about 24 hours. Then added an amount of TEA for proper gel consistency [13]. Further, the water-soluble materials and the oil-soluble materials were mixed, separately. The oil phase was added into the water phase while stirring until it forms an emulsion [14]. Then the emulsion form was poured into carbomer gel base to produce emulsion gel preparation

Table 1: Formulation of Olive Oil and Clove Oil Emulsion Gel.

Ingredients	F1 (%)	F2 (%)	F3 (%)
Olive oil	2	4	6
Clove oil	5	5	5
Carbomer	2	2	2
Liquid paraffin	5	5	5
TEA	qs	qs	qs
Tween 80	7,2	7,2	7,2
Span 80	2,8	2,8	2,8
Methyl paraben	0,18	0,18	0,18
Propyl paraben	0,02	0,02	0,02
Propylene glycol	10	10	10
Distilled water	Until 100%	Until 100%	Until 100%

2.2.2. Physicochemical Evaluation

The physicochemical evaluation involved was organoleptic, homogeneity, determination of pH value, viscosity and spreadability. Organoleptic test was carried out with observing the color, smell, and texture of the preparation, visually. Homogeneity was observed visually for the presence of the coarse particle. Determination of the pH value was carried out using a digital pH meter and the viscosity was measured using viscometer. Then the spreadability test was determined by put the preparation between two object glass which give a certain amount of load [15].

2.2.3. Stability Test

1.1.1.1. Real-time Method

Stability test in real-time method was carried out by stored under different temperature conditions (4°±2°C; 30°±2°C; 40°±2°C) and tested on its organoleptic, homogeneity, pH, and SPF value. All formulations were stored for 30 days [16].

1.1.1.2. Freeze-thaw Cycling Method

Stability test in freeze-thaw cycling method was determined by stored in the 4°±2°C for 24 hours, then moved at 40°±2°C for 24 hours and counted as one cycle. This test was held in six cycles (12 days) [17].

2.2.4. UV Protection Activity Test

Determination of the UV protection activity of sunscreen preparation was determined by calculating the SPF value in vitro using the spectrophotometer. About 0.5 grams of samples was dissolved in 100mL of 70% ethanol then was diluted to 5000ppm and sonicated about 5 minutes [18]. Each concentration of sample solutions was measured of the absorbance value at a wavelength of 290-320nm with 5nm intervals. The SPF values was obtained by the equation:

$$SPF = CFx \sum_{290}^{320} EE(\lambda)xI(\lambda)xAbs(\lambda)$$

Where EE is the erythema effect spectrum; I is the spectrum light intensity; Abs is the absorption of the sunscreen; CF is the correction factor (value is 10) [19].

3. RESULTS and DISCUSSIOn

3.1. Physicochemical Evaluation

The clove oil and olive oil emulsion gel had a thick consistency, white in colour and smell of clove. The white colour comes from the emulsification process of oil phase and water phase, as shown in Figure 1. Visually, the preparations had no coarse particle, which indicated that the clove oil and olive oil emulsion gel were homogenous. The measurement of pH value, viscosity, and spreadability are shown in Table 2.

Table 2: Physical and Chemical Characteristics of Olive Oil and Clove Oil Emulsion Gel.

Formula	pH	Viscosity (cps)	Spreadability (cm/g)
1	4.84 ± 0.08	7333 ± 250	4.03 ± 0,22
2	4.82 ± 0.06	11167 ± 624	4.03 ± 0.27
3	4.82 ± 0.09	6933 ± 94	3.91 ± 0.32

The different olive oil concentration (2%, 4%, and 6%) had no significant different in pH and spreadability value, but different in viscosity, significanty. As shown in Table 2, the higher concentration of olive oil resulted no significant different in pH value. This phenomenon occurred because the essential oil has no effect on pH value. The viscosity and spreadability value was not follow the theory that the higher viscosity, the lower the spreadability value [20].

Figure 1: Physical Appearance of Olive Oil and Clove Oil Emulsion Gel of Formula 1 (a); Formula 2 (b); and Formula 3 (c).

3.2. Stability Test

3.2.1. Real-time Method

The results of real-time stability testing of olive oil clove oil emulsion gel showed no change in colour, odor and phase separation after storage at 4˚C, 30˚C, and 40˚C. The pH values were not affected by the addition of olive oil and the storage temperature, almost in all formulas. Table 3 showed that the pH value of all preparations was not change over time, except for formula 2 in 40˚C storage.

Table 3: The pH Value in Real-time Stability of Olive Oil and Clove Oil Emulsion Gel.

Formula	1^{st} day	30^{th} day		
		4˚C	30˚C	40˚C
1	4.84 ± 0.08	4.88 ± 0.006	4.78 ± 0.07	4.89 ± 0.03
2	4.82 ± 0.06	4.85 ± 0.010	4.81 ± 0.05	4.90 ± 0.04
3	4.82 ± 0.09	4.89 ± 0.051	4.82 ± 0.06	± 0.06

3.2.2. Freeze-thaw Method

The results of freeze-thaw stability method showed that all formulas did not change organoleptically and showed no phase separation. The pH values were significantly

different, which indicated that the preparation was not stable in extreme condition. The pH value measurement results were shown in Table 4.

TABLE 4: The pH Value in Freeze-thaw Stability of Olive Oil and Clove Oil Emulsion Gel.

Formula	1^{st} day	12^{th} day
1	4.84 ± 0.08	4.48 ± 0.010
2	4.82 ± 0.06	4.50 ± 0.129
3	4.82 ± 0.09	4.47 ± 0.071

3.3. UV Protection Activity

UV protection activity was determined using the SPF value measurement. Effectiveness of sunscreens were classified by SPF value which contained in the preparation. Olive oil has an SPF value of 13.2074 and clove oil has 37.3099. In Table 5, it is known that the highest SPF value is Formula 3. The higher the added olive oil, the greater the SPF value obtained. But the stability test implies that the SPF value of olive oil and clove oil emulsion gel was not stable at storage, significantly.

TABLE 5: UV Protection Activity (SPF Value).

Formula	1^{st} day	Freeze-thaw	Real-time		
			4°C	30°C	40°C
1	20.91 ± 0.29	2.59 ± 0.12	1.91 ± 0.05	10.87 ± 0.15	2.79 ± 0.23
2	22.52 ± 0.52	2.53 ± 0.13	2.07 ± 0.03	9.66 ± 0.48	3.29 ± 0.76
3	23.40 ± 0.45	3.89 ± 0.48	2.28 ± 0.06	9.07 ± 0.02	2.95 ± 0.18

4. CONCLUSION

The 6% of olive oil and 5% clove oil in emulsion gel showed the best formulation due to their physicochemical, stability, and UV protection activity. It showed SPF value of 23.40 ± 0.45. It indicates that this preparations are included in the medium category protection of sunscreen and have the potential as a sunscreen product. We suggest for room temperature storage for this preparation.

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