Formulation of sunscreen cream from *Eucheuma cottonii* and *Kaempferia galanga* (zingiberaceae)

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Abstract. The objective of this research was to obtain the best formulation *E. cottonii* and *K. galanga* for the production of sunscreen cream. The ratio of *E. cottonii* and *K. galanga* was 1:1, mixed in three versions of a cream with concentrations of 10%, 20%, and 30%. The sunscreen cream were evaluated based on the antioxidant activity, The sun Protection Factor (SPF) activity of the cream quality (viscosity, pH, homogeneity), and the microbial test. The IC50 values of the sunscreen cream were 44.59 μg/mL (for cream with 10% concentration), 31.47 μg/mL (for cream with 20% concentration), and 20.32 μg/mL (for cream with 30%). The SPF values were 8.8 (for cream with 10% concentration), 13.3 (for cream with 20% concentration), and 16.7 (for cream with 30% concentration). The viscosity values were 19,400 cps (for cream with 10% concentration), 18,000 cps (for cream with 20% concentration), and 15,600 cps (for cream with 30% concentration). The pH values of sunscreen cream were 6.52 (for cream with 10% concentration), 6.61 (for cream with 20% concentration), and 6.75 (for cream with 30% concentration). All of the sunscreen cream treatments were of a good homogeneity and absent at microbial test. The best formulation was Cream D, so that the *E. cottonii* and *K. galanga* have synergistic effects to be used as sunscreen.

Keywords: cosmetics, seaweed, sunscreen cream

1. Introduction

One of the solar radiation is ultraviolet light. Exposure to ultraviolet light has a negative effect on human skin. Ultraviolet (UV) is divided into UVA (320-400 nm), UVB (320-290 nm) and UVC (100-290 nm) [1]. Among all, UV B is the most hazardous to human skin, and causes even skin cancer after long-term exposure. However, the intensity of UVB exposure less than UVA [2].
Protection from UV light can potentially be provided by sunscreens. A good sunscreen is derived from natural ingredients such as seaweed and \textit{K. galanga} \cite{3-4}. The bioactive compounds found in seaweed are very prospective to be applied in sunscreen cream. Seaweeds are a good source of some water-soluble vitamins (B1, B2, B12, and C) \cite{5}, fat-soluble vitamins (β-carotene with vitamin A activity, and vitamin E), antitumor, anticoagulant, and antioxidant activity \cite{6-9}. \textit{E. cottonii} is reported to have antioxidant and protective effects against UV-induced ROS degeneration in keratinocytes. They have K-carrageenan \cite{10}, flavonoid, and phlorotannins \cite{11}.

\textit{K. galanga} is a plant belonging to the Zingiberaceae family and which contains the most ethyl-cinnamate and ethyl-para-methoxycinnamate. The most dominant content in \textit{K. galanga} is ethyl-p-methoxycinnamate (25.96% to 87.4%) \cite{12}. The potency of the rhizome (\textit{K. galanga}) includes its anti-inflammatory, analgesic, nematicidal, antimicrobial and antioxidant properties \cite{13-16}. \textit{K. galanga} has been used as medicines, such as traditional medicine.

Very little information is available on the combination of \textit{E. cottonii} and \textit{K. galanga} for sunscreen cream. It has not been widely practiced, even though both are very useful for health. Therefore, \textit{E. cottonii} and \textit{K. galanga} can be a great combination for application in cosmetic products. The objective of this study was to obtain the best formulation of sunscreen cream from \textit{E. cottonii} and \textit{K. galanga}.

2. Materials and Methods

2.1. Materials

The rhizomes of \textit{K. galanga} were collected from Cikande District and the seaweed of \textit{E. cottonii} was collected from Lontar Beach on August 2016. Both areas were in Serang, Banten, Indonesia. Both of the materials were authenticated in Bogor Agricultural University, Bogor, Indonesia.

2.2. Sample preparation

\textit{E. cottonii} was washed and soaked for 12 hours with demineralized water. \textit{E. cottonii} that was separated from the demineralized water was mashed using a blender with the addition of demineralized water (1:1), resulting in homogeneous seaweed \cite{17}. The rhizomes were at first cut into small pieces and then sun-dried for ten consecutive days. The pieces were then dried by oven at 60°C for 24 hours. Finally, the dried crisps were ground into a coarse powder using a grinding machine \cite{18}.

2.3. Sunscreen cream formulation

The composition of the cream formula consisted of two phases, the oil phase and water phase (table 1). The oil phase components included stearic acid (Merck), liquid paraffin (Merck), emulgide (Merck), and cetyl alcohol (Merck). The water phase components included glycerin (Merck), triethanolamine (Merck), and aquades. Additives used were methylparaben (Merck), butylated hydroxytoluene (Sigma-aldrich), fragrance (Salsa), seaweed extract (\textit{E. cottonii}), and \textit{K. galanga} powder \cite{19}.
Table 1. Preparation of the sunscreen cream formulas [19].

| Materials               | Cream A (%) | Cream B (%) | Cream C (%) | Cream D (%) |
|-------------------------|-------------|-------------|-------------|-------------|
| Oil Phase               |             |             |             |             |
| Emulgide®               | 8           | 8           | 8           | 8           |
| Stearic acid            | 4           | 4           | 4           | 4           |
| Cetyl alcohol           | 2           | 2           | 2           | 2           |
| Liquid paraffin         | 2           | 2           | 2           | 2           |
| Water Phase             |             |             |             |             |
| Glycerin                | 2           | 2           | 2           | 2           |
| Triethanolamine (TEA)   | 1           | 1           | 1           | 1           |
| Aquades                 | ad. 100     | ad. 100     | ad. 100     | ad. 100     |
| Additives Fragrance     | q.s         | q.s         | q.s         | q.s         |
| Methyl paraben          | 0.2         | 0.2         | 0.2         | 0.2         |
| Butylated hydroxytoluene| 0.1         | 0.1         | 0.1         | 0.1         |
| Seaweed extract (E. cottonii) | 0       | 5           | 10          | 15          |
| K. galanga. powder      | 0           | 5           | 10          | 15          |

Trademarks (mixture of cetyl stearyl alcohol and alkyl polyglycol ether as the base for the cream)

2.4. Antioxidant activity

A sample weighing as much as 10 mg was dissolved in 1 mL dimethyl sulfoxide. A 200 μL extract solution was plated into microplate, and 100 μL of DPPH (Sigma-aldrich) solution with 125 μM in ethanol p.a was added. The mixture was homogenized and then incubated at 37°C for 30 minutes in a dark room. The result was measured with a microplate reader (IWAKI) at 517 nm wavelength. Control positive used was an ascorbic acid with the concentration of 1.25-20 ppm [20, 21]. Ethanol was used as a negative control. DPPH radical inhibition activity (%) can be calculated by the formula:

\[
\% \text{ Inhibition} = \frac{\text{absorbance control} - \text{absorbance sample}}{\text{absorbance control}} \times 100\%
\]  

2.5. SPF activity

The sunscreen efficacy was determined by SPF value using a UV spectrophotometry (Shimadzu UV-1800) [22]. Every sample (400 mg) was dissolved in 25 mL ethanol (Sigma-aldrich). The curve used had a wavelength of 290-320 nm, with 5 nm interval [23]. The value of EE x I was constant [24]. The value data can be seen in table 2.

Table 2. Value data of EE x I for SPF measurement.

| Wavelength (λ) nm | EE x I  |
|------------------|---------|
| 290              | 0.0150  |
| 295              | 0.0817  |
| 300              | 0.2874  |
| 305              | 0.3278  |
| 310              | 0.1864  |
| 315              | 0.0839  |
| 320              | 0.0180  |
| Total            | 1.0000  |
The absorbance data was calculated using the Mansur equation:

\[
SPF = CF \times \sum_{\lambda=290}^{320} EE(\lambda) \times I(\lambda) \times \text{abs}(\lambda)
\]  

Information:
- \(CF\) = correction factor
- \(EE\) = spectrum of erytermal effects
- \(I\) = spectrum of intensity from the sun
- \(\text{Abs}\) = absorbance of the sample

2.6. Evaluation of the sunscreen cream
The evaluation of the sunscreen cream included:

1. Determination of viscosity using viscometer (Brookfield DV-E) at 100 r/min, with the spindle No. 7 [19].
2. Determination of pH using pH meter (CP-401 Elmeiron) that was first calibrated. The pH meter was calibrated using a standard buffer solution. The cream (0.5 gr) was dissolved in 50 mL of distilled water and its pH was measured [25].
3. The formulations were tested for the homogeneity by judging their visual appearance and touch affinity [19].

2.7. Microbial test
The total microbial analysis on cream consisted of making a PCA (Merck), BFP (butterfield's phosphate buffered) reagent solution (Merck), and total microbial test [17].

3. Results and discussion

3.1. Antioxidant activity of the sunscreen cream
Knowing the antioxidant activity in cream formulas is very important to determine the effectiveness of a product. A compound as an antioxidant was very strong, strong, medium and weak if the IC_{50} value were less than 50 μg/mL, between 50-100 μg/mL, between 100-150 μg/mL, more than 150-200 μg/mL, respectively [20]. Cream formulas from \(E.\) cottonii and \(K.\) galanga provided the synergistic effect, shown in table 3. All of the treatments had very strong antioxidant values, and the best formulation for sunscreen cream was Cream D (20.32 μg/mL), which was appropriate because the higher concentration had stronger activity [21]. The antioxidant values of cream A (0% concentration), cream B (10% concentration), cream C (20% concentration), and cream D (30% concentration) were 280.60 μg/mL, 44.59 μg/mL, 31.47 μg/mL, and 20.32 μg/mL, respectively.

The antioxidant activity was mainly due to the total phenolic content and flavonoids [26], including luteolin and apigenin from \(K.\) galanga [16]. \(K.\) galanga contains ethyl p-methoxycinnamate, which has been reported to inhibit monoamine oxidase [27]. The antioxidant activities of leaves and rhizome extracts have been reported to be 77 μg/mL and 17 μg/mL [28]. Seaweed has a phenolic component and contains antioxidants that are able to fight free radicals by donating one or more electrons to free radicals [29]. The percentages of DPPH free radical scavenging activity by \(Kappaphycus\) alvarezii (\(E.\) cottonii) extracts ranged from 18.34 to 35.63% with the total phenolic compound (TPC) in the range of 6.74 to 17.32 mgGAE/100 g of wet weight sample [30].
Table 3. Evaluation of E. cottonii and K. galanga sunscreen cream.

| Sample       | IC₅₀ activity (µg/mL) | SPF   | Viscosity (cP) | pH    |
|--------------|-----------------------|-------|----------------|-------|
| Cream A (0%) | 280.60 ± 8.00         | 0.80 ± 5.10  | 39,000         | 7.42  |
| Cream B (10%)| 44.59 ± 12.22         | 8.80 ± 6.30  | 19,400         | 6.52  |
| Cream C (20%)| 31.47 ± 2.23          | 13.30 ± 7.40 | 18,000         | 6.61  |
| Cream D (30%)| 20.32 ± 6.23          | 16.70 ± 8.80 | 15,600         | 6.75  |

3.2. SPF activity of the sunscreen cream

The effectiveness of a sunscreen is usually expressed in sun protection factor (SPF). To be effective in preventing sunburn and other skin damages, a sunscreen product should have a wide range of absorbance, between 290-320 nm [22]. Cream D (16.70) was very good for counteracting UV rays because it has a high SPF value compared to other treatments. The SPF value of cream A (0%), cream B (10%), cream C (20%), and cream D (30%) were 0.80, 8.80, 13.30, and 16.70, respectively (table 3).

The main bioactive compounds from plants to be used in cosmetics are phenolic and flavonoid [26]. Phenolic compounds are able to fight free radicals caused by ultraviolet radiation. Flavonoids have three photoprotector properties, namely ultraviolet light absorption, antioxidant properties, and modulation of several DNA signaling pathways [31]. Red algae contain cyclohexylamine chromophore, which can absorb ultraviolet light. The presence of mycosporine-like amino acids (MAAs) in red algae has a very high potential in absorbing UV-A rays [32]. Besides phenolic and flavonoid in K. galanga, apigenin is a compound that can absorb ultraviolet light. The bioactive compound from K. galanga extract is reported to be non-toxic and non-irritant for the body [15].

3.3. Evaluation of the sunscreen cream

3.3.1. Viscosity. Viscosity governs the many properties of the product, such as spreadability and pourability of the product from the container [33]. The viscosities of the cream were in the range of 15,600-39,000 (table 3) centipoises, which indicated that the cream was easily spreadable by small amounts of shear.

3.3.2. The pH. The pH of creams was determined to examine the possible side effects of acidic or alkaline pH, which can lead to irritation of skin. Acidic or alkaline pH may cause irritation to the skin and influence the rate of hydration of polymer. The cream in general has a pH 6-9 for sunscreen [33]. The pH formulation of the sunscreen cream was in the range 6.52 to 7.42. Cream A (0%) has the highest pH value and cream B (10%) has the smallest.

3.3.3. Homogeneity. All formulations produced a uniform distribution of extracts in the cream. This was confirmed by visual appearance and by touch. Therefore, all formulation was of a good homogeneity.

3.4. Microbial test

Microorganisms can grow if there is water content in the product and a lipolytic process which causes odor. Microbial contamination in pharmaceutical preparations can reduce the quality of the preparation with changes in color, odor, color turbidity, and pH [17]. The results of a total microbial assay on cream A, B, C, and D showed no microbial colonies, which means that the cream was safe from microbes and was in accordance with the standards required. That was due to the presence of methylparaben and other substances that caused no microbes to grow [19, 34].
4. Conclusion

The best formulation of the sunscreen cream from *E. cottonii* and *K. galanga* was cream D (30%). Sunscreen cream formulations from *E. cottonii* and *K. galanga* have a synergistic effect, and need to be developed further.

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