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Utilization of seaweed porridge *Sargassum* sp. and *Eucheuma cottonii* as cosmetic in protecting skin

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Abstract. UV radiation has negative effects on the health of the skin. The use of sunscreen everyday can absorb at least 85% sunlight at a wavelength of 290-320 nm. The purpose of this research was to find the best formulation of seaweed porridge *Sargassum* sp. and *Eucheuma cottonii* as a sunscreen cream qualified by antioxidant activity, SPF, physical evaluation of cream analysis and stability analysis. Cream formulation comprised of 5%, 6%, 7% and control. The best percentage of stocks from cream *E. cottonii* and *Sargassum* sp. were the cream preparation C. The value of antioxidant activity and SPF were 185 ± 0.02 mg/mL and 2.1988, respectively. The pH value belongs to alkaline. Cycling test showed the cream preparation of the slurry *E. cottonii* and *Sargassum* sp. remained stable, there was no phase separation and discoloration. Centrifugal test results indicate that the cream has a shelf life for 1 year due to the absence of phase separation after given effect centrifugal force with a speed of 3,800 rpm for 5 hours.

Keywords: antioxidant, SPF, sunscreen, UV-A, UV-B

1. Introduction

The skin quality reflexes the health and appearance of human life. Cleanliness and beauty of the skin are very important, especially for women [1]. Various attempts were made to obtain a clean skin, bright, not dull, and free of blemishes. Being white and bright is a concept of beauty and a dream for many people, especially for Asian women [2]. Exposure to UV-A rays stimulates the formation of melanin in the dermis layer that works as a protective layer on the skin. The UV radiation approximately 300 nm (UV-B) penetrate the layers of the stratum corneum and epidermis causes erythema. Exposure to UV rays changes the skin connective tissue. Long-term exposure to ultraviolet gives the effect of dull skin tone, darker and black spots appearance [3]. The daily use of sunscreen can absorb at least 85% of sunlight at a wavelength of 290-320 nm [4].

Seaweed is one of the commodities whose production increased by 20.9% in 2013 of 7.5 million tons and in 2014 targeted 10 million tons [5]. Seaweed contains a source of water-soluble vitamins (B1, B2, B12, C) and fat-soluble (β -karaoten with the activity of vitamin A and E). Vitamin E is a fat-



soluble vitamin in seaweed which contains much antioxidant activity [6]. *Euchema cottonii* contains phycocyanin mycosporine acid (Maas) consisting of imine derivatives containing UV chromophore aminocycloheximine absorber [7]. *Sargassum* sp. had higher levels of the average - average ascorbic acid as much as 49.01 ± 0.75 mg/100g [8]. *Sargassum* sp. has greater antioxidant activity than other types of *Caulerpa racemosa*, *Ulva lactuca* and *Gracilaria tenuistipitata* with IC₅₀ values of each of 1.08 ± 0.83 , 15.05 ± 0.61 , 103.73 ± 0.59 , 24.22 ± 0.87 µg/mL [9].

Sargassum sp. contains phlorotannin compounds belonging to brown algae polyphenols, formed from phloroglucinol (1,3,5-trihydroxybenzene) [10]. Red, green and brown seaweed produce various bioactive compounds such as antibacterial, antioxidant. In addition, it can be used as a pigment source (food coloring/dye) [11-19]. The use of seaweed as a source of antioxidants has been previously evaluated in cosmetics [20-26]. Seaweed can also be processed into seaweed salt extract, which can be used for hypertensive patients [27].

Bioactive components found in seaweed is very prospective for cosmetics because it contains terpenoids, carotenoids, and polysaccharides (fucoidan, carrageenan, alginate, agar), polyunsaturated fatty acids, and amino acids [28]. Carrageenan has been widely used in various industries of food, medicine, textiles, and cosmetics because it can be an emulsifier, thickener, stabilizer, and gelling. Sunscreen formulations in this study will use *Sargassum* sp. and *E. cottonii* in dosage forms slurry. In addition for economical, the dosage form slurry tends to be cheaper and safer than chemicals. Products made also been marketed in five provinces in Indonesia through the activities of Micro, Small and Medium Enterprises (MSMEs) are implemented by the Ministry of Affairs Marine and Fisheries.

Active compounds from the raw materials and the seaweed *Sargassum* sp. and *E. cottonii*, and vitamin E is known of *Sargassum* sp. amounted to 165.19 mg/mL and *E. cottonii* 160.01 mg/mL. The antioxidant activity of *Sargassum* sp. and *E. cottonii* of the methanol extract of 57.05 mg/mL and 105.04 mg/mL and contains active components including flavonoids, phenols hydroquinone, and triterpenoids which allegedly is a potential compound used as a raw material sunscreen cream [23]. So, the research is necessary to study. The purpose of this study was to get sunscreen formulations that meet the requirements through an antioxidant test, sun protection factor (SPF) test, and evaluation of physical and stability test.

2. Materials and Methods

The main materials used were a red seaweed *E. cottonii* from Serang, Banten and brown seaweed *Sargassum* sp. was from Kepulauan Seribu, Jakarta. Emulgate, cetyl alcohol, liquid paraffin, TEA, glycerin, xanthan gum, GMS, distilled water, fragrance. The main tools used in this study is digital scales (Tanita KD-160), an analytical balance type 210-LC (Adam, United States), UV-Vis spectrophotometer - 1601 (Shimadzu, Japan), pH meter type 510 (Eutech Instrument, Singapore), homogenizer (Omni-Multimix Inc., Malaysia), penetrometer (Herzoo, Germany), centrifugation (Kubota 5100, Japan), oven (Mettler, Germany), and glass tools.

2.1. Preparation of seaweed

E. cottonii seaweed washing done using saline solution. *E. cottonii* bleaching process by soaking solution of CaO [29]. Sample rate and a solution of 1:20 [30]. After that *E. cottonii* and of *Sargassum* sp. were mixed with distilled water (ratio 1:1 w/w) using a blender.

2.2. Preparations of cream

The preparation of the cream followed previous research [31] with some modifications. Seaweed porridge added with emulgate, cetyl alcohol, liquid paraffin, and nipagin. The mixture was heated until the temperature reaches $\pm 80^{\circ}\text{C}$ and called the oil phase. TEA, glycerin, xanthan gum, GMS, seaweed *Sargassum* sp. and *E. cottonii* heated until $\pm 80^{\circ}\text{C}$ and called liquid phase. The oil phase incorporated into the liquid phase when both mixtures reached $\pm 80^{\circ}\text{C}$ until cream is formed then added some fragrance.

2.3. Analysis of cream

Antioxidant activity was carried out with the DPPH method. The method was based on the ability of the sample used in reducing DPPH stable free radicals. Analysis SPF using Spectrophotometer UV-vis. Samples were taken as much as 1 gram in each sample, dissolved in 95% ethanol as much as 100 mL mixed until homogeneous. As much 5 mL of the solution is transferred to the flask measure and add ethanol to 25 mL. Previously the spectrophotometer was calibrated using 96% ethanol, the way 1 mL of ethanol is put in cuvet then the cuvette is entered in a UV-Vis spectrophotometer for the calibration process. The next step is to make test absorption curves in cuvettes, with wavelengths between 290-350 nm, use ethanol 96% as blank then set the average uptake (Ar) at intervals of 10 nm. Absorbance results recorded, then calculated the SPF value [32], the measurement of pH value stocks cream using a pH meter. The pH meter is calibrated before use. Calibration is done by using a buffer solution of pH 4 and pH 10. The pH examination is done by dipping the electrode into 1 gram of cream preparation which is diluted with distilled water up to 10 mL [33], consistency using a penetrometer. Preparations to be examined are entered into in a special container and put on penetrometer table. Consistency check conducted at week 0 and week 12 with storage at room temperature [34], evaluation cream made with organoleptic and physical testing homogeneity [35], analysis of temperature stability in the early stages for cream preparations included cycling tests and centrifugal test [36].

3. Results and Discussion

3.1. Antioxidant activity for *Sargassum* sp. dan *E. cottonii* cream

The antioxidant activity of porridge seaweed *Sargassum* sp. and *E. cottonii* using DPPH free radical methods. DPPH is a radical scavenging compound having purple color. In the reduced form, DPPH has no color. DPPH was selected due to the simplicity, quick and reagent saving. Vitamin C was used as a standard [37].

The cream C had the highest antioxidant activity with IC_{50} value 185 ± 0.02 mg/mL, however it was still in a weak category. The higher the concentration of seaweed porridge added, the higher the antioxidant activity. Antioxidant activity is considered high when the IC_{50} value is less than 50 ppm, medium for IC_{50} between 50-100 ppm, and weak if IC_{50} is 150-200 ppm [38]. The IC_{50} values of the creams are shown in table 1.

Table 1. Antioxidant activity of cream.

Cream formulation	IC_{50} value
A (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 5%)	240 ± 0.03
B (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 6%)	196 ± 0.02
C (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 7%)	185 ± 0.02
D (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 0%)	340 ± 0.01

3.2. Sun protection factor (SPF)

The effectiveness of a sunscreen cream is determined from SPF which describes the ability of a sunscreen to protect the skin from erythema [32]. The SPF values of the creams are presented in table 2. The effectivity of the sun cream can be divided into minimum, extra and ultra based on their SPF values [39].

Table 2. SPF value of creams with seaweed porridge *Sargassum* sp. and *E. cottonii*.

Cream Formulation	SPF
Cream A	2.1988
Cream B	2.3356
Cream C	2.7883
Cream D (control)	2.0982

3.3. Cream evaluation

The physical evaluation included observation organoleptic (color, odor, texture). Homogeneity test by observing the particle distribution of cream sandwiched by two glass objects. Measurement of pH using a pH meter and flow properties with a Brookfield viscometer. Test of the consistency was carried out using the penetrometer. Testing of the average globule diameter was using optical microscope in 40 times magnification.

The cream is a system that has a surface free energy of particles dispersed [35]. The results of the initial evaluation of all creams on storage (week-0) obtained cream is soft, easy applied, forming a semi-solid consistency, and easily spread on the skin. Overall results demonstrate cream A, B, C, and D have the organoleptic appearance of white and homogenous. The observation of the creams during week 0 is shown in figure 1.



Figure 1. Organoleptic observations of creams at week-0.

The addition of *Sargassum* sp. and *E. cottonii* showed no effect of discoloration and odor during the cream preparation. Homogeneity test showed all marked with a homogeneous cream in glass particles evenly dispersed objects. Figure 2 shows the homogeneity observation of cream.



Figure 2. Homogeneity observation of cream.

The cream consistency at week-0 were formula A 322×10^{-1} mm, B 320×10^{-1} mm, C 315×10^{-1} mm, D 310×10^{-1} mm. The penetration rate meets the criteria of cream preparations that feels easily to be applied and spread on the skin. The nature of the flow rate of four creams that pseudoplastic thixotropic where the cream has a lower consistency on each force per unit area (rate of shear) indicating a breakdown of structures that do not form again immediately if the stress is removed or reduced. The higher the viscosity, the higher the cream consistency as well, so that the number of cone penetrometer penetration depth is lower [40].

The pH values measured from the cream formula A was 8.48, cream B was 8.47, cream C was 8.45 and cream D was 6.45 at week-0. The fourth cream showed a pH value which leaned toward a weak base. The pH value which can be tolerated by the skin is 4.2 to 6.5 [1].

3.4. Stability of creams

3.4.1. *Storage of cream at temperature of $7\pm 2^\circ\text{C}$, $27\pm 2^\circ\text{C}$ and $40\pm 2^\circ\text{C}$.* The results showed A, B, C and D creams stored at low temperature $7\pm 2^\circ\text{C}$, room temperature $27\pm 2^\circ\text{C}$ and high temperature $40\pm 2^\circ\text{C}$ during storage week 2 and week 4, did not show changes in color, odor, texture and phase separation did not occur. Observations on creams of porridge *Sargassum* sp. and *E. cottonii* (A, B, C) as well as the control cream was tested on the storage temperature of $7\pm 2^\circ\text{C}$, $27\pm 2^\circ\text{C}$, and a temperature of $40\pm 2^\circ\text{C}$ is shown in figure 3. The pH value of the cream is presented in table 3.

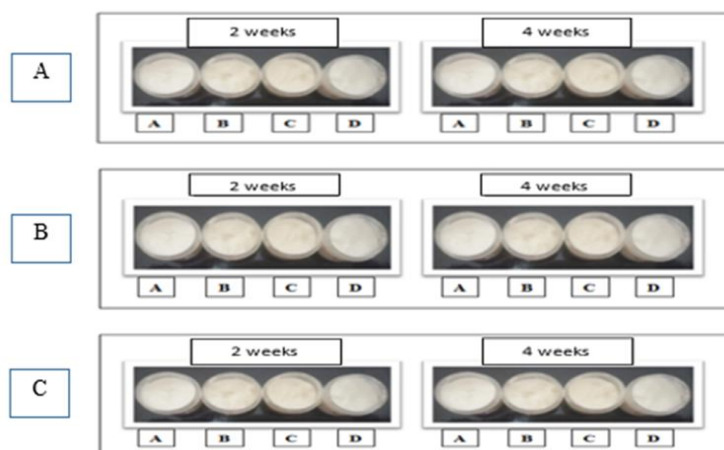


Figure 3. Organoleptic observations on cream at a temperature (a) $7\pm 2^\circ\text{C}$, (b) $27\pm 2^\circ\text{C}$ and (c) $40\pm 2^\circ\text{C}$ after 2 weeks and 4 weeks of storage. A: cream *Sargassum* sp. and *E. cottonii* 5%; B: cream *Sargassum* sp. and *E. cottonii* 6%; C: cream *Sargassum* sp. and *E. cottonii* 7%; D: cream without added *Sargassum* sp. and *E. cottonii* (control).

Table 3. Degree of acidity (pH) of the cream at week 2 and 4.

Temperature	Cream Formulation	Week	
		2	4
$7\pm 2^\circ\text{C}$	A (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 5%)	8.37 ± 0.12	8.26 ± 0.01
	B (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 6%)	8.38 ± 0.09	8.27 ± 0.18
	C (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 7%)	8.44 ± 0.21	8.42 ± 0.20
	D (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 0%)	8.44 ± 0.11	8.38 ± 0.15
$27\pm 2^\circ\text{C}$	A (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 5%)	8.28 ± 0.05	8.19 ± 0.10
	B (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 6%)	8.28 ± 0.13	8.16 ± 0.09
	C (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 7%)	8.27 ± 0.12	8.19 ± 0.13
	D (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 0%)	8.28 ± 0.11	8.17 ± 0.13
$40\pm 2^\circ\text{C}$	A (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 5%)	8.39 ± 0.15	8.25 ± 0.10
	B (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 6%)	8.38 ± 0.12	8.28 ± 0.18
	C (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 7%)	8.33 ± 0.14	8.21 ± 0.19
	D (<i>Sargassum</i> sp. and <i>E. cottonii</i> cream 0%)	8.36 ± 0.02	8.27 ± 0.07

The pH values that can be tolerated by the skin are range from 4.2 to 6.5. Measurement of pH value is very important to know the level of acidity of the cream preparation. The degree of acidity for cosmetic products or products that are used for external use, which are directly in contact with the skin, should be in accordance with the pH balances. The recommended pH of cream products ranges from 4.5 to 8.0 [41]. If the cosmetic product has a pH value is very high or very low will cause skin irritation [34].

3.5. Cycling test

The percentage of emulsion stability can be calculated if the occurrence of phase separation in an emulsion after a freeze-thaw cycle [42]. A cycling test was carried out to test the product for the

possibility of crystallization and as an emulsion test on cream as an indicator of emulsion stability [43]. Emulsifying ingredients such as stearic acid, glycerol, cetyl alcohol and tri ethanol amine (TEA) were able to unify the oil phase and water phase in the cream preparation so that the cream could be homogeneously mixed and remain stable at temperature changes [44]. Observations of the cycling test can be seen in table 4.

Table 4. Cycling test observations.

Cream formulation	Beginning of cycle	End of cycle
Cream A (5%)	No phase separation	No phase separation
Cream B (6%)	No phase separation	No phase separation
Cream C (7%)	No phase separation	No phase separation
Cream D (control)	No phase separation	No phase separation

Centrifugal test was investigated by the physical stability semisolid dosage. Stokes Law indicates the formation of the cream is a function of gravity and increase in gravity to accelerate phase separation. The effect of centrifugal force provided by centrifugation with a speed of 3800 rpm for 5 hours is considered equivalent to the effects of gravity to be received cream in storage for a year. The centrifugation test results showed no phase separation in the A, B, C, D creams and the cream remained stable. The results of mechanical tests can be seen in figure 4.

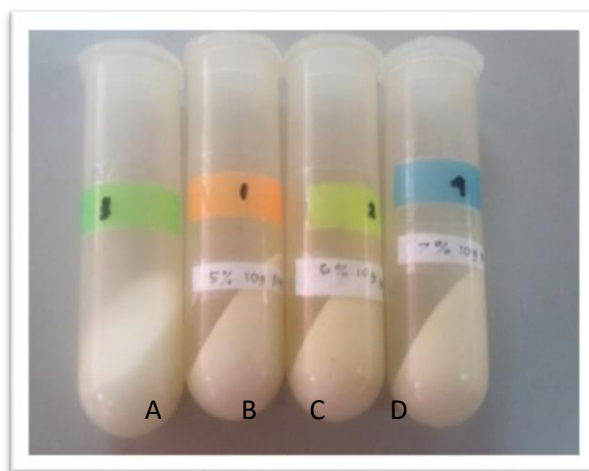


Figure 4. Observation of creams after centrifugal test. A: cream *Sargassum* sp. and *E. cottonii* 5%; B: cream *Sargassum* sp. and *E. cottonii* 6%; C: cream *Sargassum* sp. and *E. cottonii* 7%; D: cream without added *Sargassum* sp. and *E. cottonii* (control).

4. Conclusion

The best preparation of cream stock was cream C. The values of antioxidant activity and SPF were 185 ± 0.02 mg/mL and 2.1988. The pH value belongs to alkaline condition. Cycling test showed the cream preparation of the slurry *E. cottonii* and *Sargassum* sp. remained stable, there was no phase separation and discoloration. Centrifugal test result was predicted that the cream has a shelf life for 1 year due to the absence of phase separation after given effect centrifugal force with a speed of 3,800 rpm for 5 hours.

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