**Characteristics of seaweed porridge Sargassum sp. and Eucheuma cottonii as raw materials for lip balm**

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**Abstract.** A lip balm is needed to overcome skin problems such as dry lips, cracked, dark, and wrinkled and premature aging of the lips. A lip balm that contains antioxidants and bioactive compound is needed to inhibit free radical activity and overcome problems in the lips. Seaweed is a natural material that is quite abundant and has a variety of bioactive compounds, including seaweed Sargassum sp. and E. cottonii. This study aimed to characterize and obtain the best formulas of seaweed Sargassum sp. and E. cottonii combination in producing lip balm preparations, as well as obtaining the best lip balm formulations perceived from antioxidant analysis, pH, and loss on drying. This research consists of two stages of research. First stage research was to characterize and prepare the seaweed raw material, characterize and production of seaweed Sargassum sp. and E. cottonii porridge. Second stage research was the preparation of lip balm preparations. The results showed that the best ratio of seaweed Sargassum sp. and E. cottonii was found on 1:1 ratio with IC₅₀ antioxidant value of 576.41 ppm, pH 5.39, and Loss on drying 3.52%. The pH value of the lip balm in corresponds to the SNI and pH of the normal human skin balance.

**Key words:** antioxidant, E. cottoni, lip balm, Sargassum sp., and seaweed porridge.

1. **Introduction**

Lips are organs susceptible to free radicals exposure. One function of the lips is where the entry of food and beverages every day in a fairly large frequency. Food and drink may contain substances that can damage the lips [1]. The physical condition of the lips are susceptible to problems from the outside, so the lips need of course both from within and from outside the body [2]. The use of lip balm containing antioxidant agents can help the damage to the lip’s skin from free radicals. The ultraviolet (UV) rays in sunlight can damage the lips. UV rays can cause the lips to become dry, cracked, burned, swollen or arise brown spots, dark, and wrinkled on the lips [3].

Lipstick or lip balm cosmetic products that have antioxidant activity are available in the market with various brands, but people are concerned about the uses of synthetic antioxidant materials in these cosmetic preparations. Antioxidant compounds can be produced from synthetic and natural compounds. Synthetic antioxidant compounds include butyl hydroxy toluene (BHT), ascorbic acid, kojic acid, mercury and hydroquinone [4]. Kojic acid and butyl hydroxy toluene (BHT) are carcinogenic when use in high concentrations and can cause damage to the skin. Use of mercury in
cosmetics is harmful, because mercury is carcinogenic [5]. Another alternative is to use natural antioxidant ingredients that are safer which is derived from seaweed [6].

Indonesia is the second country after China in seaweed production in 2013, which is 34% of 26,896,004 tons, which produces the world [7]. The production of brown seaweed in Indonesia is also quite large around 482,400 tons/year [8]. According to the Ministry of Marine Affairs and Fishery seaweed production in Indonesia in 2015 reached 10.2 million tons/year. Because of the high production it is needed more effort to optimize the use of seaweed as an active ingredient in cosmetic products including lip balm.

Seaweed Sargassum sp has antioxidant activity of 1.08 ± 0.83 μg/mL, greater than Caulerpa racemosa, Ulva lactuca and Gracilaria sp. with IC₅₀ values of 15.05 ± 0.61; 103.73 ± 0.59, and 24.22 ± 0.87 μg/mL respectively [9]. Antioxidant activity Sargassum sp. and E. cottonii from ethanol extract showed IC₅₀ value of 57.05 μg/mL and 105.04 μg/mL [10]. The results of bioactive components such as flavonoids, hydroquinone phenols and triterpenoids which are potential compounds used as raw materials in cosmetics [11]. According to [12] seaweed Sargassum polycystum contains flavonoids, steroids, and triterpenoids. Sargassum sp. contains phlorotamin compounds that are polyphenols in brown algae, but are not found in terrestrial plants. Sargassum sp. has a median ascorbic acid level of 49.01±0.75 mg/100 g [13]. Seaweed E. cottonii contains protein, lipids, carbohydrates, vitamin C, α-tocopherol, and minerals can be used as a growth medium of lactic acid bacteria [14]. The bioactive components present in seaweed are highly prospective in cosmetics because they contain terpenoids, carotenoids, and polysaccharides (fucoidan, carrageenan, alginate, agar), compound unsaturated fatty acids, and amino acids [15].

Carrageenan has been used in many food industries, medicine, textiles and cosmetics, because it is permanent as an emulsifier, thickener, stabilizer, and gelling [16]. Seaweed E. cottonii has the potential as a natural antioxidant, because it contains an active component of flavonoids that act as antioxidants [17]. Making cosmetics from natural ingredients today only use the preparation in the form of extracts of active ingredients. The use of extract preparations may affect production costs and higher product selling prices, and it is feared that the remaining chemical solvent residue of extraction and waste products, both from the solvent used and the solids from the extracted material. Research conducted using seaweed is processed in the form of doses of porridge safer, economical, environmentally friendly, zero waste, and easily adapted by the community. Further research is needed on the characterization of seaweed porridge used as a lip balm preparation. The aim of this research was to get the characteristic and ratio of seaweed grass Sargassum sp. and E. cottonii best in producing lip balm preparations, as well as the best lip balm formulations seen from antioxidant, pH, and loss on drying analyzes.

2. Materials and method

2.1. Materials

The materials used in this research were seaweed E. cottonii and Sargassum sp., emulglade, cetyl alcohol, vaseline, cocoa butter, glycerin, methyl paraben, fragrance, purified water, demineralization moisture, 1,1-diphenyl-2-picylhydrazyl (DPPH), ethanol (Merck), vitamin C, olive oil, beeswax, alcohol, ethanol, meyer reagent, dragendorff, and wagner, chloroform, NH₃, H₂SO₄, NaOH, HCl, amyl alcohol, FeCl₃, anhydride. The analytical devices used in this research are spectrophotometer (Optima type SP-300), measuring cup, blender, vortex, knife, container, thermometer, cutting board, volumetric pipette, micropipet, test tube, porcelain cup, electric cooker, stirring rods, analytical scales, incubators, digital scales, ovens, pH meters (Lutron YK-2001 PH), moisture bath.

2.2. Methods

2.2.1. Sample collection. Seaweed samples are the main raw material is brown seaweed Sargassum sp. obtained from the Pasauran island, Umbul Village Tanjung Cinangka District, Serang Regency, Banten and red seaweed E. cottonii obtained from the cultivation of people in Lontar, Tirtayasa District, Serang regency, Banten. The samples obtained were washed and dried by way of aerated
[18]. Fresh seaweed was washed using sea moisture to remove dirt, moss, mud, and sand. Samples are dried in a place protected from direct sunlight to avoid damage to bioactive compounds [19].

2.2.2. Characterization of raw materials. *E. cotonii* and *Sargassum* sp. were immediately dried after taken out from the ocean. All seaweeds were chemically characterized including moisture content test and rehydration weight.

2.2.3. Seaweeds porridge preparation [20]. Seaweeds *Sargassum* sp. or *E. cotonii* each weighed as much as 100 g. After weighing the sample washed, then soaked for 12 hours in demineralized water. The comparison between seaweed and demineralized water is 1:20. Both seaweeds were then washed clean using demineralized water to remove dirt, salt, and sand that still attached to the seaweed. Seaweed porridge mixture was a sample ratio 1 and 1 demineralized moisture, then homogenized with a blender. Seaweed porridge preparation, performed percentage analysis of development (rehydration), moisture content, pH, and phytochemical test.

2.2.4. Lip balm formulation (Modification of Maharany et al. 2017). The process of making lip balm as a lip moisturizer includes ingredients that fall into the oil phase i.e. olive oil, cocoa butter, Vaseline, emulgade, cetyl alcohol. The ingredient of moisture phase was glycerin and moisture. The additional material used was the seaweed *Sargassum* sp. and *Eucheuma cottonii*, methyl paraben, and strawberry fragrance. The mixing phase of oil and moisture phase is carried out at 75°C. Base lip balm formed added seaweed porridge *Sargassum* sp. and *E. cottonii* with different ratio (1: 1), (1: 2), (2: 1) as much as 30%, control (without seaweed) and methyl paraben.

2.3. Analytical methods

2.3.1 Moisture content analysis [21]. Moisture content analysis was conducted to determine the amount of moisture contained in the sample of seaweed *Sargassum* sp. and *E. cotonii*. The first stage was to dry the porcelain cup in the oven at 105°C to obtain a constant weight for 1 hour. The cup was placed into a desiccator (approximately 15 minutes) and allowed to cool and then weighed. Seaweed samples *Sargassum* sp. and *E. cotonii* were weighed 5 g, then the filled bowl of the sample was put into the oven at 105°C for 5 hours. The cup is inserted into the desiccator and allowed to cool and then weighed.

2.3.2. Measurement of pH [22]. Weighing 3 g samples, diluted with 10 mL of distilled moisture, did the pH measurement in the sample. The pH measurements of the samples were performed directly by dipping the pH meter electrode into the diluted sample until the pH value appeared on the pH meter screen (YK-2001PH brand Lutron) stable.

2.3.3. Phytochemical test [23]. Phytochemical tests were performed to determine the content of bioactive compounds contained in the sample. The samples were tested in the form of seaweed porridge. Phytochemical tests performed include testing of alkaloid compounds, flavonoids, saponins, tannins, hydroquinone phenols, and steroids/triterpenoids on seaweed *Sargassum* sp. and *E. cottonii*.

2.3.4. LoD (Loss on Drying) analysis [24]. Porcelain cup weighed and recorded the empty weight. Lip balm samples were weighed into petri dishes. The LoD test is done by drying the dry sample at 105°C for two hours, then the cup is removed and stored in the desiccator. The percentage of LoD is calculated by the formula:

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\text{LoD} (%) = \frac{\text{Initial sample weight} - \text{final sample weight}}{\text{Weight of initial weight}} \times 100\%
\]
2.3.5. **Antioxidant activity test** [25]. Testing of antioxidant activity using DPPH method (free radical damping method). Antioxidant activity test includes making DPPH stock, vitamin C stock, sample stock, blank and activity test using DPPH method. Percentage of free radical activity inhibitors is obtained from sample absorbance values. The linear regression equation is obtained from the relationship between the sample concentration and the percentage of inhibition of free radical activity. The antioxidant activity of each sample and the antioxidant comparable to vitamin C was expressed by percent of inhibition.

3. **Result and discussion**

3.1. **Raw materials moisture**

Moisture content analysis is intended to determine the free water content contained in dried seaweeds Sargassum sp. and E. cottonii. Moisture content greatly affects shelf life, acceptability, physical properties and freshness of the sample [26]. The result of moisture content analysis of sample of Sargassum sp. and E. cottonii were 31,32% and 38,71%. The results of moisture content of Sargassum sp. dryness obtained higher than that of [27], which is 12.37%. The moisture content of dried E. cottonii obtained was higher than that of [14], which was 14.34%. The drying process can cause this difference in moisture value. Drying process is carried out i.e dry wind; the material is not directly exposed to sunlight. The longer the drying time, the moisture content of a food will be lower.

3.2. **pH Sargassum sp. and E. cottonii porridge**

The pH measurement of the Sargassum sp. and E. cottonii was conducted to determine the degree of acidity of the seaweed porridge used as the raw material in the preparation of lip balm. Results of pH analysis on seaweed grass Sargassum sp. and E. cottonii have a pH of 6.91 and 6.57, respectively. This pH value indicates that the Sargassum sp. and E. cottonii is safe to use as cosmetic raw material (lip balm) because it has pH balance and in accordance with SNI number 16-4399-1996. The value of degree of acidity for cosmetic products or products used topically is 4.5-7 pH of cosmetic products for the skin according to SNI number 16-4399-1996 suggested ranged from 4.5-8.0 [28]. The pH value of neutral seaweed pH is due to having been soaked using demineralized moisture. The demineralized moisture pH is in the pH range of 6.5 to 7.5.

3.3. **Active components of Sargassum sp. and E. cottonii**

Phytochemical analysis performed was alkaloids, steroids/triterpenoids, flavonoids, saponins, hydroquinone phenols, and tannins. Phytochemical compounds detected on the Sargassum sp. including steroids/triterpenoids, flavonoids, saponins. Alkaloid content, hydroquinone phenol and tannins were not detected in the preparation of seaweed grass Sargassum sp. reseach. Phytochemical compounds detected in the preparation of E. cottonii slurry were only alkaloid content, while the other compounds were undetectable. The bioactive components found in seaweed are highly prospective for use in cosmetics [11].

3.4. **pH lip balm**

Products that have a lower pH than physiological pH of the skin will cause an irritant reaction, if it has a higher pH than physiological pH of the skin will cause dry skin of the lips cracked. The pH value for cosmetic products is 4.5-7 [28]. The result of analysis of variance (ANOVA) showed that the difference of Sargassum sp. and E. cottonii have an effect on pH value of lip balm produced at α = 0.05 (Appendix 6). Duncan test results showed that the highest pH lip balm value at 2: 1 ratio is significantly different from the ratio of 1: 1 and 1: 2. The pH value of lip balm in this study ranged from 5.05 to 5.65. These results are in accordance with the physiological pH of the skin of the lips. The physiological pH of the lip skin is 4.2-5.6 [29]. Normal skin has a more acidic pH. SNI No. 16-4399-1996 pH recommended skin products range from 4.5-8.0 [40]. According to [28], the ideal topical cosmetic is not irritate the skin of the lips. The likelihood of irritation on the skin of the lips is very large when the preparation is too acidic or too alkaline.
3.5. Loss on Drying (LoD)
Loss on Drying (LoD) analysis on lip balm was performed to determine the ability of lip balm in maintaining moisture, judging by the weight of shrinkage after heated using oven at 105°C for 2 hours. The result of analysis of variance (ANOVA) showed that the difference of Sargassum sp. and E. cottonii have an effect on the value of loss on drying (LoD) of lip balm produced at α = 0.05 (Appendix 7). Duncan test results showed that the highest loss on drying (LoD) lip balm at 2: 1 ratio is significantly different from the 1: 1 and 1: 2 ratio. The value of Loss on Drying (LoD) in lip balm has a value between 3.01-4.52%. The highest value of LoD was found in lip balm control (without adding seaweed) of 4.52% and the lowest in lip balm with a 1: 2 treatment of 3.01%. The lower the weight of the shrinkage after heating the material is able to hold moisture longer and can act as a moisturizer.

Lip balm with the addition of seaweed E. cottonii has less shrinkage weight, this is because seaweed E. cottonii has a hydrocarbon in the form of carrageenan. [30] stated that the carrageen from seaweed E. Cottonii shows the ability to spread and have moisture holding capacity (WHC) so it can be used as a moisturizer. According to [31], polysaccharides in seaweed have a high moisture retention ability (WHC) than cellulose fibers. According to [32], hydrocolloids in alginate form derived from seaweed Sargassum sp. can reduce moisture loss and dehydration during storage.

3.6. Antioxidant Activity
Antioxidants are compounds capable of inactivating oxidation reactions by preventing the formation of free radicals, because excessive oxidation reactions can trigger the formation of free radicals [33]. Antioxidant activity can be determined by IC$_{50}$ value. Analysis of variance (ANOVA) showed that the difference of Sargassum sp. and E. cottonii had an effect on IC50 antioxidant value of lip balm produced at α = 0.05 (Appendix 8). Duncan test results showed that the best IC$_{50}$ antioxidant lip balm value at 1: 1 ratio was significantly different with the ratio of 1: 2 and 2: 1. Lip balm formula with the best IC$_{50}$ value is on the ratio of seaweed grass Sargassum sp. and E. cottonii 1: 1 of 576,41 ppm. The lowest antioxidant activity was on lip balm control (without addition of seaweed porridge) with IC$_{50}$ value of 1261,78 ppm. Lip balm with the addition of seaweed porridge has better antioxidant activity, when compared to lip balm control. Seaweed porridge contains an active compound in the form of flavonoids that have antioxidant activity. Lip balm with the addition of Sargassum sp. and E. cottonii have weak antioxidant activity because they have IC$_{50} >$ 200 ppm. IC$_{50}$ values contained in a material are closely related to the bioactive compounds contained in the material [34]. A compound is said to be a very powerful antioxidant if the IC$_{50}$ value is less than 50 ppm, strong for IC$_{50}$ between 50-100 ppm, while if IC$_{50}$ is worth 100-150 ppm and weak if IC$_{50}$ is worth 150-200 ppm [25]. Seaweed has a phenolic component and contains antioxidants capable of fighting free radicals by donating one or more electrons to free radicals. Polyphenol compounds are a group of heterogeneous and structural compounds classified as flavonoids, phenolic acids, and lignin [35]. Polyphenolic compounds are good antioxidants and polyphenol derivatives of seaweed have been explored as functional food [36]. Factors affecting antioxidant activity include: storage, heating, oxygen, and irradiation, which can lead to chain initiation and propagation of the oxidation reaction, thereby decreasing the antioxidant activity in the material [37].

4. Conclusion
The ratio of seaweed grass Sargassum sp. and E. cottonii for the best lip balm formula is 1: 1 with IC$_{50}$ antioxidant value of 576.41 ppm, pH 5.39, and Loss on Drying 3.52%. The pH value of lip balm corresponds to the SNI and pH of the normal human skin balance. Seaweed grass Sargassum sp. and E. cottonii contain phytochemical compounds such as steroids, triterpenoids, flavonoids, and saponins. The content of phytochemical compounds indicates that Sargassum sp. and E. cottonii potential to be used as natural antioxidant source in making lip balm.
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