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Seaweed extract (*Sargassum polycystum)* as a preservative on sunscreen cream with the addition of seaweed porridge

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Abstract. The use of sunscreen is highly recommended to protect the health of the skin from ultraviolet radiation. Most commercial sunscreens contain artificial preservatives such as methylparaben, which can be detrimental to health. Preservatives in sunscreen can be substituted with natural ingredients, in which *Sargassum polycystum* ethanol extract is offered as an alternative in this research. The preservative is made from *S. polycystum* extract with addition of seaweed porridge of 1:1 *S. polycystum* and *Eecheuma cottoni* mixture. The quality of cream is tested using total microbial test, durable power prediction, antioxidant and SPF value. Its physical stability is tested through sensory testing, pH measurement, cyling test and centrifugal test. Butylated hydroxytoluene (BHT) is used as control and is then treated with methylparaben and the extract for comparison, the extract was found to have comparable microbial results with 8 weeks of preservation compared to methylparaben (9 weeks). In addition, the cream shows stability up to one year, a balanced pH according to SNI, shows no foul odor, antioxidant activity (IC_{50}) of 105.42, SPF value of 2.00, and moderate to favorable consumer acceptance.

Keywords: antibacterial, preservative, *Sargassum polycystum*, sunscreen

1. Introduction

Seaweed that grows in Indonesian waters is recorded around 555 species. One of the potential seaweed as a cosmetic raw material and its abundant availability is *Eucheuma cottonii* and *Sargassum* sp. Food and Aquaculture Organization (FAO), reported that Indonesia was the second country after China in seaweed cultivation production in 2013 which amounted to 34% of the 26,896,004 tons produced by the world [1]. Public awareness of the importance of skin health care is a driving factor for increasing demand for skin care cosmetic products. The use of skin care cosmetic products is one of the efforts to protect the skin from the negative effects of weather conditions. Air humidity in Indonesia can reach 80% with temperature [2]. Continuous sun radiation will cause black spots to appear. Sunscreen cream products have been widely developed and produced on the market, but there are some concerns including the use of hazardous chemicals that do not meet the specifications of the products produced with those on the label. Cosmetics generally contain a mixture of chemical compounds and not many come from natural sources [3]. The demand for cosmetics from herbal ingredients is currently growing very rapidly [4]. This expansion is due to the availability of raw materials from nature. Raw materials for aquatic products that have the opportunity to be developed into cosmetic products are seaweed. *Sargassum* sp. have greater antioxidant activity compared to *Caulerpa racemosa*, *Ulva lactuca* and *Gracilaria* types in the case of IC_{50} with 1.08±0.83, 15.05±0.61, 103.73±0.59, 24.22±0.87 µg/mL respectively [5]. *Sargassum* sp. is a type of brown algae

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that is able to absorb UV light [6]. *Sargassum* sp. contains fucoidan and phenolic components that are able to capture free radicals [7].

Red seaweed *E. cottonii* contains phycocyanin compounds which contain mycosporine acid (MAAs) and consists of imine derivatives containing aminocycloheximine UV absorbent chromophore [8]. Red seaweed contains antioxidant compounds that can inhibit the penetration of strong UV light into tissues or cells. In addition, *E. cottonii* antioxidant activity showed IC_{50} of 105.04 μg/mL [9]. The active components produced include flavonoids, phenol hydroquinones and triterpenoids which are thought to be potential compounds used as raw materials for sunscreen creams.

2. Materials and Methods

2.1. Materials and tools

The main ingredients used were *S. polycystum* seaweed and *E. cottonii*. Ingredients for making creams were emulgade cream, stearic acid, methyl paraben, liquid paraffin, butyl hydroxy toluene (BHT), glycerin, triethanolamine (TEA), and fragrance. The material used for the analysis were aquades, NaCl, ethanol 96%, CaO, 2.2 diphenyl-1-picrylhydrazyl (DPPH) powder, Plate Count Agar (PCA), Nutrient Broth (nb), and alcohol 70%. The tools used in this study were digital scales, analytic scales, spectrometers, pH meters, centrifuges, ovens, glassware (Pyrex), stirrers, blenders, aluminum foils, incubators, counters, bunsen, spray bottles, sterile plastic, hot plate, Erlenmeyer, and serology pipette, mask, and gloves.

2.2. Sampling

E. cottonii used for porridge in a dry form and at a maximum harvest age of 45 days. *S. polycystum* seaweed was taken in the waters of Banten Bay, Serang Regency, Banten Province. *S. polycystum* seaweed was cleaned and sorted from sand or objects that are carried during the picking process and washed with sea water. Seaweed that has been washed with sea water, then rinsed with fresh water that flows to remove the salt or sand content that is attached. The seaweed was then stored in a styrofoam container in the transport process to the laboratory. *S. polycystum* was extracted by single maceration method using ethanol to obtain its active compound and its yield was determined.

2.3. Antibacterial activity test

Antibacterial activity testing included the stages of preparation of test bacterial rejuvenation, identification of test bacteria and testing stages. The stages of test bacterial rejuvenation preparation were a modification of a standard reference from NCCLS or the National Clinical Laboratory Standards Committee which included the manufacture of PCA solid media, the manufacture of NB liquid media and using *Staphylococcus aureus* bacteria and *Escherichia coli* as standard test bacteria [10].

2.4. Preparation of Sargassum and E. cottonii seaweed porridge

Preparation referred to the previous research [11]. The process of making *S. polycystum* porridge. Carried out through three stages: washing, soaking and draining. *S. polycystum* seaweed. Those that had been washed and dried were rinsed then soaked for 12 hours using distilled water. *S. polycystum*, the 12 hours soaked then drained. *S. polycystum* porridge, done by mixing *S. polycystum*, and distilled water, then homogenized using a blender.

E. cottonii seaweed preparation was done through four stages, namely washing, bleaching, soaking and draining. *E. cottonii* washing was done with running water to get clean seaweed from foreign objects such as sand, wood, twigs and sticking dirt. *E. cottonii* bleaching was done to get a white and attractive appearance. Blanching using a combination of distilled water and lime (CaO) 0.5% for 30 minutes while continuing to knead to help speed up the bleaching process. *E. cottonii* was rinsed again and continued the process of soaking *E. cottonii* with distilled water for 12 hours. The next process was making porridge by mixing *E. cottonii* and aquades, then homogenizing it using a blender.

2.5. Making sunscreen cream preparations

The ingredients used in making cream preparations were divided into two phases. Oil-soluble ingredients include emulate, liquid paraffin, stearic acid, and BHT dissolved until homogeneous at a temperature of ±75°C called oil phase (preparation 1). Simultaneously, water-soluble ingredients include glycerin, TEA, distilled water, and 1:1 *S. polycystum* and *E. cottoni* seaweed slurry preparations dissolved until homogeneous at ±75ºC called water phase (preparation 2). After that (preparation 1) and (preparation 2) homogenized and reached the same temperature ±70ºC, mixing was done until it was formed (preparation 3) in the form of a homogeneous cream [12].

In this study modification was carried out by replacing methyl paraben with extract of *S. polycystum* as preservative in the cream of sunscreen seaweed. Then do (preparation 3) with 3 treatments. The first treatment was given the addition of methyl paraben and BHT, the second treatment was given an addition of *S. polycystum* and BHT extracts, and the third treatment was only added with BHT (control) and then homogenized for ±10 minutes at 40ºC. Fragrance was put into 3 treatments of the cream preparation and homogeneous for ±3 minutes.

3. Results and Discussion

3.1. Sampling

Sampling was done by diving directly at the sea in the depth of 30-100 cm from the coastline. *S. polycystum* was then cleaned and sorted from sand or objects that are carried during the picking process and washed with sea water. Seaweed that has been washed with sea water then dried and stored in a styrofoam container and the extraction method for *S. polycystum* carried out in this study is single maceration using ethanol.

In the maceration process the crude extract of ethanol from the evaporation results in a characteristic paste-like solvent with a distinctive aroma. In color characteristics, the relative color was brownish, the yield was 2.73% in the first replication and 3.96% in the second replication. The percentage of yield produced from macroalgae extraction using ethanol solvents ranged from 2-3%. Based on these data it can be identified that *S. polycystum* contains active compounds which are relatively soluble in polar solvents.

3.2. S. polycystum antibacterial activity

The antibacterial activity can be seen by inhibitory zones (figure 1). Test inhibitory zones formed for *S. aureus* bacteria were found at each concentration, namely at concentrations of 50 µg/mL (5.3 mm), 100 µg/mL (5.8 mm), 200 µg/mL (5.11) and 300 µg/mL (6.19), and inhibitory zones for *Escherichia coli* bacteria were found at concentrations of 100 µg/mL (5.11 mm), 200 µg/mL (5.18 mm) and at concentrations of 300 µg/mL (6.5 mm).

A B

Figure 1. Exposure of inhibitory zones to *E. coli* bacteria (A) and *S. aureus* (B).

The difference in antimicrobial activity can be caused by the susceptibility of each bacterium. Bacteria have a vulnerability to different physical facilities and chemicals. The resistance of

microorganisms to several types of antibiotics can be caused by the properties possessed by the microorganisms themselves.

3.3. Preparation of E. cottonii and S. polycystum

Porridge Preparations *S. polycystum* slurry preparation through washing, soaking and draining processes, but not through bleaching. The taking of *S. polycystum* was done directly by diving, so that *S. polycystum* was used fresh. Fresh seaweed has a higher antioxidant activity compared to dry seaweed [13].

The immersion process of *E. cottonii* and *S. polycystum* was carried out for 12 hours to remove residual lime (CaO) in the bleaching process of *E. cottonii*. Another goal of immersion is to produce gel on seaweed and ensure the seaweed is clean before making a slurry preparation. The slurry preparation was made by homogenizing between seaweed and distilled water with a ratio of (1:1) using a blander.

3.4. Durable power test

The total microbial testing results showed that microbial colonies found in cream preparations from week 0 to week 4 were still below the total microbial limit so that the cream could be used. The total microbial limit in the cream required by SNI 16-4399-1996 is a maximum of 1.0x102 colonies/gram.

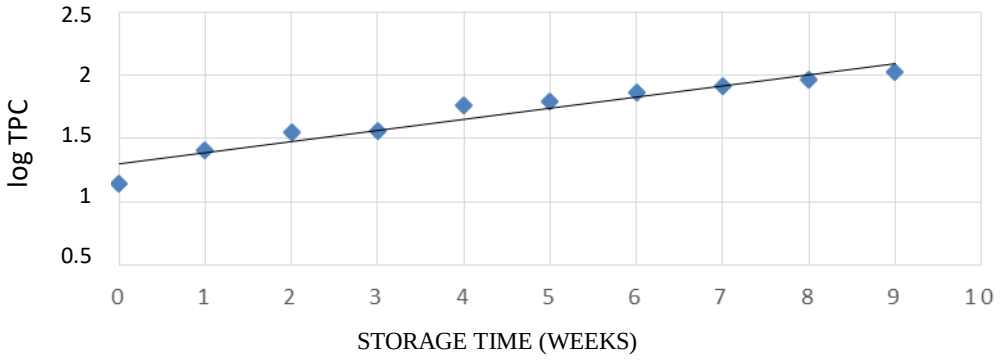

Figure 2. TPC linear regression graph treatment of methyl paraben + BHT.

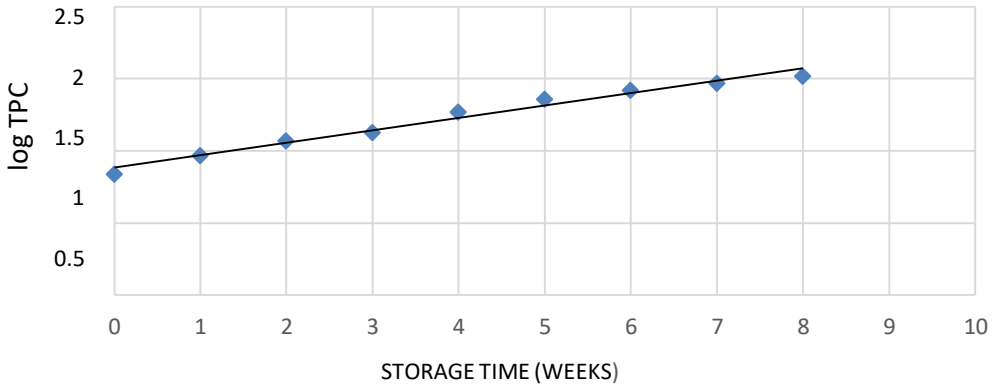

Figure 3. TPC linear regression + BHT linear regression graph.

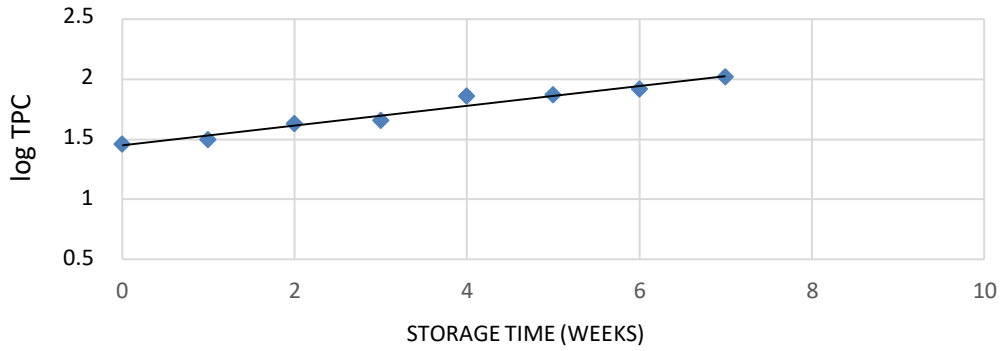

Figure 4. TPC linear regression graph for BHT treatment.

Based on the linear graph (figure 2-4), the results of the TPC test can be predicted that at 7 weeks cream with BHT treatment was rejected, which means that it does not meet the standard, while the extract treatment + BHT was rejected at week 8 and methyl paraben + BHT was rejected at 9 weeks. That the use of methyl paraben preservatives and *S. polycystum* extracts could inhibit microbial activity in cream compared to non-preservative cream.

3.5. Antioxidant activity

Antioxidant activity of seaweed sunscreen cream + BHT treatment was able to reduce free radicals with a concentration of 50%, with IC_{50} values of 105.42. The addition of slurry *E. cottonii* and *S. polycystum*, as well as *S. polycystum* extracts were thought to have an influence on the IC_{50} value of cream preparations. Seaweed has a phenolic component and contains antioxidants that can fight free radicals by donating one or more electrons to free radicals [14]. Free radicals are atoms or molecules that are very unstable and reactive and damage the tissue [15].

The smaller IC_{50} value shows the greater antioxidant activity in the test material. A compound is said to be a very strong antioxidant if IC_{50} values are 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.

3.6. SPF sunscreen cream value

The value of Sun Protection Factor (SPF) shows the effectiveness of a sunscreen cream in protecting the skin. The results of the calculation of the SPF value of sunscreen cream preparation are presented in table 1.

Table 1. The results of calculating the SPF values of sunscreen creams.

Cream	SPF value
Methylparaben+BHT	1.00
Extract+BHT	2.00
BHT	1.00

The classification of the sunscreen ability level is minimum if the SPF is between 2-4, 4-6 moderate, 6-8 extra, 8-15 maximum, and ultra if the SPF is more than 15 [16]. The extract has a SPF value of 2.00 which is still in the range of moderate sunscreen protection, thus it is potential to use in sunscreens.

3.7. Sensory test

Sensory testing is a subjective test that is implemented with the value of consumer preference for product acceptance. Sensory testing used a hedonic scale with untrained panelists aged 20-35 years totaling 30 people. The results of sensory test can be seen in figure 5.

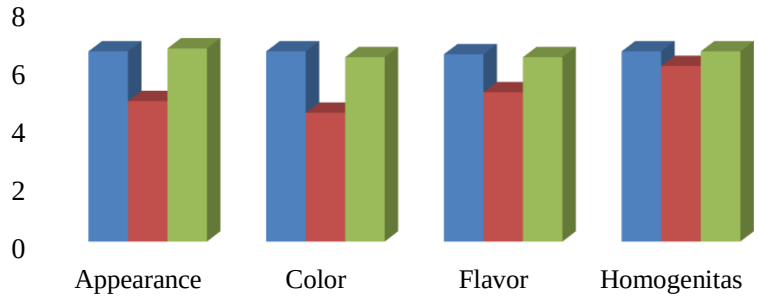

Figure 5. Average value of sensory testing, : methylparaben+BHT, : extract+BHT, : BHT.

From this figure, we can conclude there is no significant difference between cream with extract and cream with methylparaben in sensory testing. It is safe to assume that the extract is potential to replace methylparaben as an additive.

3.8. Appearance

The sensory results showed that the panelists' response to the appearance of sunscreen cream gave different ratings. It can be concluded that the cream + BHT extract sample obtained the lowest value of 4.8, because the cream extract + BHT had a less attractive appearance, while the panelists' favorite value on the appearance of the cream sample methyl paraben + BHT and BHT ranged from 6.5 to 6.6 which means that the panelists give an assessment between normal and somewhat like.

3.9. Color

Color is one of the parameters of visual observation attached to a product. Color can be one of the assessment factors in the selection of a product by consumers. Panelists' preference for the cream color of methyl paraben + BHT and BHT samples ranged from 6.3 to 6.5, which means the panelists gave a rather favorable assessment. Whereas the + BHT extract sample had the lowest preference value because the addition of *Sargassum polycystum* extract caused the cream color to dark green.

3.10. Flavor

A pleasant and easily recognizable aroma will generally be preferred compared to an unrecognized aroma. Fragrance or fragrance can duplicate the desired aroma [17]. The higher the percentage of aromatic compounds, the longer the intensity and aroma are obtained. Panelists' favorite score on the aroma of the cream ranged from 5.1 - 6.4 which means that the panelists gave an assessment between rather like to like.

3.11. Homogeneity

Homogeneity is a parameter to see the effectiveness of evenly or not mixing ingredients in the product. The panelists' preference value for the cream homogeneity of methyl paraben + BHT and BHT samples was 6.5, which means the panelists gave a rather favorable assessment, while the cream + BHT cream sample had the lowest value of 6 because the addition of extract made extract + cream BHT is more dense than methyl paraben + BHT and BHT cream.

3.12. The pH of sunscreen cream preparations

The results of pH measurements for 10 weeks at different temperatures are at low temperatures (4±2°C), room temperature (28±2°C), and high temperatures (40±2°C). The pH value of the cream addition of seaweed porridge with three treatments namely methyl paraben + BHT cream, extract + BHT and BHT has increased with the length of storage time, but still in the range of skin pH balance value and in accordance with SNI Number 16-4399-1996.

3.13. Cyling test

Cycling test tests were carried out in two different conditions, namely at low temperatures of 4±2ºC and high temperatures of 40±2ºC for 6 cycles or 12 days. Cycling tests were conducted to test the

product for the possibility of crystallization and as an emulsion test on cream as an indicator of emulsion stability [18]. The results of the cycling test showed that there was no phase separation in the cream emulsion, discoloration and no change in aroma the oil phase and the water phase in the cream preparation so that the cream can be mixed homogeneously and remain stable at extra temperature changes, the color changes that occur in the cream preparation are due to the oxidation process of vitamin C. The oxidation process will occur faster if the cream is exposed by air, light and hot. After experiencing the color oxidation process the cream can turn into a bit darker.

3.14. Centrifugal test

Centrifugation was carried out at a speed of 3,800 rpm for 5 hours. The results of observation after centrifugation testing showed that seaweed cream preparations with methyl paraben + BHT treatment, extract + BHT and BHT. No color and odor changes or phase separation. The absence of phase separation in cream preparations is due to the surfactant used to protect oil droplets on the cream preparation so that the oil phase and water phase are well mixed [16].

4. Conclusion

Seaweed (*S. polycystum*) extract with the addition of seaweed porridge has potential to replace methylparaben as an adequate natural preservative in sunscreen cream.

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