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FULL PAPER

Development of peel-off mask ethanolic extract sargassum polycystum and its antioxidant activity using DPPH (2,2-diphenyl-1-picrylhydrazyl) method

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One of the unique characteristics of the peel-off mask is that it will create a layer of translucent, elastic film. After drying, there is no residue and it may be removed without rinsing. The production of masks from natural materials is preferable to that from synthetic materials, which might have negative side effects and alter natural contour of the skin. *S. polycystum* is one of the natural products that can be used as a mask with natural ingredients. Antioxidants included in *S. polycystum* act as a deterrent to free radicals. *S. polycystum* contains phenolic compounds, carotenoids, laminarins, alginates, fukoidans, florotanins, and ectoin among other antioxidants. The objective of the study was to find the optimal amount of *S. polycystum* extract to incorporate into peel-off masks with high antioxidant activity. The sample is prepared using the *Ultrasonic Assisted Extraction* technique with *S. polycystum* powder and ethanol as the solvent. A peel-off mask was made utilizing *S. polycystum* extract in concentrations of 0%, 3%, and 4%. Testing the mask's quality includes an organoleptic test, a pH test, a homogeneity test, a stability test, a drying test, and an antioxidant activity test using DPPH with an IC_{50} concentration. *S. polycystum* extract can be made into a peel-off mask, according to the research. The formulation of a 4% *S. polycystum* extract peel-off mask was selected since it met the requirements of a peel-off gel mask of the Indonesian National Standard (it is homogeneous and stable, pH 5.6, and dry time 17 minutes with an IC_{50} of 125.20 μg/mL, which is a moderate antioxidant).

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Introduction

The skin serves as the body's outermost layer of defense against various external threats. The damaging impacts of pollutants, sun exposure, and free radicals can all result in skin damage [1-8]. As molecules, free radicals have atoms with one or more unpaired electrons on their outer orbit, making them relatively unstable. If they attach to electrons from other molecules,

free radicals stabilize. An antioxidant is a chemical substance that can transfer one electron to a free radical, hence suppressing free radicals. An oxidation reaction happens when free radicals are present. Antioxidants are necessary to decrease the surplus of free radicals. Even in very modest amounts, antioxidants can prevent the oxidation process [9-16].

Sargassum polycystum

type of seaweed that is a member of the *brown algae* family, is one plant that may be useful as an antioxidant. Because seaweed includes a number of vitamins and minerals that the skin needs, it is thought to provide aesthetic benefits [17]. Secondary metabolite, particularly phenolic compounds with significant levels of bioactivity antioxidants, is found in *brown algae*. In addition, it includes terpenoids, which can be employed as antioxidants [18-20]. Alkaloids, polyphenols, tannins, and flavonoids are also present in *Sargassum polycystum* [21].

The main components of the antioxidant chemicals produced by Phaeophyceae are thought to be the phenol compounds and their derivatives [22]. Additional sources of antioxidants found in brown algae include carotenoids, laminarins, alginates, fukoidans, florotanins, and phenolic compounds [19]. According to previous research, the 96% *S. polycystum* ethanol extract possesses antioxidant activity with an IC_{50} value of 492 µg/ mL [23]. *S. polycystum* has the potential to be used as a raw material in cosmetic industry. According to prior studies, 13% of the ethanolic extract of *S. polycystum* is ectoin [24]. Ectoin has been commercially marketed as an ingredient in a number of over-the-counter medicines and is one of the most prevalent and potent cytoprotectants [25,26]. The effects of UVA-induced and accelerated skin aging are also resisted by ectoin at several cell levels [27]. In addition, it helps to stabilize cell membranes, reduce inflammation, and reduce DNA damage brought on by UV, infrared, and visible light [28].

Compared to plants grown on land, the use of marine plants in cosmetics is less developed. Furthermore, the presence of ectoin makes it better for use in cosmetic ingredients. The use of mask has many benefits not only refreshing, repairing, and tightening of facial skin, but also improving blood circulation, stimulating the activity of skin cells, lifting dead skin cells, softening the skin, and providing nutrient on the skin [29].

The *peel-off* gel mask is one of the alternative solutions that can increase the user's convenience [30]. A peel-off face mask has the advantage of preventing dry, dull, and wrinkled skin on the face as well as the ability to reduce UV-induced pores [31]. Once the peel-off gel mask dries, it may be removed straight away without needing to be rinsed, effectively eliminating dead skin cells [29]. Due to the occlusiveness of the polymer layer created, *peel-off* gel masks can improve the effects of the main ingredient (active ingredients) on the epithelium area of the skin while also increasing skin hydration [30]. The peel-off gel mask has also an advantage in that it provides combed hair a cool taste and dries quickly by generating a layer of washable film.

The peel-off gel mask has crucial components, mainly film-forming and gelling agents. In this study, carbomer served as the gelling agent and PVA as the film-forming agent. The peel-off effect is produced by polyvinyl alcohol. In the meantime, carbomer is a substance that thickens gels by absorbing liquids and causing them to be held and create a gel mass [32]. Carbomer was selected above alternative gelling agents because it is a synthetic polymer that creates a more transparent system, better viscosity, simple dispersion in water, and greater release consistency [33,34]. Using an occlusion and tensor action mechanism, PVA can smooth the skin after drying [35,36]. It is safe to use methylchloroisothiazolinone and methylisothiazolinone as preservatives in the

formulation of cosmetic products designed to be rinsed off or stayed on [37].

The aim of this study was to design and conduct the best formulation for the development of peel-off mask antioxidants from *Sargassum polycystum* extract and to reduce the use of harmful chemicals that might harm the skin on the face. For antioxidant activity, a DPPH test will be run to measure the mask's antioxidant capacity.

Experimental

Materials and methods

Sargassum polycystum was obtained from Sumenep, Madura Island. Other materials are included Polyvinyl alcohol (PVA), Carbomer, Xanthan Gum, Polyvinylpyrrolidone (PVP), coloring, aquadest, 1,3 butylenglycol, glycerine, Triethanolamine (TEA), and preservative (methylchloroisothiazolinone and methylisothiazolinone) ordered from Tritunggal Arthamakmur, Indonesia. Ethanol was from Merck, Indonesia.

A. Sample preparation

Sargassum polycystum powder was weighed and extracted using 96% ethanol in an Erlenmeyer at a ratio of 1:20 with *Ultrasonic Assisted Extraction* method, and then extracted for 3x10 minutes with a 5-minute pause. The filtrate was separated and evaporated using a rotary evaporator [38].

Because ethanol is a solvent that is both universal and selective in dissolving the intended chemical compounds and more effective in breaking down non-polar cell walls to produce the desired chemical compounds, it was used for the extraction in this work [39].

B. Peel-off mask formulation

Carbomer was developed into aquadest, and then added TEA. After that, it was adjusted till pH 6.5 (A) and xanthan gum 2% was taken to A and blended. Thereafter, it was placed in a 70-80 °C water bath (B). PVA and PVP were mixed (C). C was inserted into xanthan gum solution (B). Coloring and aquadest were mixed, and then the desired color (D) was adjusted. After that, D was inserted into C solution. The extract was dissolved into ethanol, and then glycerin and 1.3 butylenglycol were added until homogeneous. Thereafter, they were added to (D) and mixed. Likewise, preservatives was added and mixed until homogenous. The formulation can be seen in Table 1.

TABLE 1 Formulation of peel-off mask

No.	Materials	F0(%)	F1(%)	F2(%)
A	Aquadest	56.5	56.5	55.2
	Carbomer	0.5	0.5	0.8
B	Xanthan gum 2%	5	5	5
C	PVA	6	6	6
	PVP	2	2	2
D	Coloring	0.6	0.6	0.6
	Water	2.6	2.6	2.6
E	1,3 butylenglycol	1.5	1.5	1.5
	Glycerine	1.0	1.0	1.0
	S. polycystum extract	-	3.0	4.0
	Ethanol	20	20	20
F	Methylchloroisothiazolinone	0.2	0.2	0.2
	Methylisothiazolinone	0.2	0.2	0.2
G	Vanilla	0.5	0.5	0.5
H	TEA	0.1	0.1	0.1
	Total	100	100	100

C. *Characteristic of peel-off mask*

This test included organoleptic of mask, pH, homogeneity of peel off mask, and drying time. Organoleptic testing was performed by observing the peel-off mask's color, shape, scent, and consistency. A pH meter was used to check the pH test. The test for drying time was applying one gram of the preparation to the arm skin, drying time was tracked using a recording clock until the mask's film layer forms [27]. Homogeneity was observed by weighing as much as 1 g of sample and applied to a clean, dry object glass to form a thin layer. The glass of the object was then covered with the prepared glass. The peel-off gel mask showed a homogeneous arrangement when no rough details are visible, the texture appears flat and does not crumble [24].

D. Antioxidant activity using DPPH assay

In a 100 mL volumetric flask, 1 g of sample was weighed, added to ethanol, and thoroughly mixed, and then 1 mL DPPH and 3 mL ethanol p.a. were added to 1 mL of the sample solution before homogenizing. For 30 minutes, the test solution and the control solution were incubated at 37 °C. A UV-Vis spectrophotometer was used to detect the test solution's absorption at a wavelength of 517 nm.

Results and discussion

The *Ultrasonic Assisted Extraction* technique is still being used in this study. Ultrasonic extraction is a potential extractive technique since it allows for the comprehensive extraction of active plant components with very little energy expenditure, a shorter processing time, and greater safety [40].

The formation and implosion of gas microbubbles in the liquid's center are caused by pressure fluctuations brought on by the waves produced during ultrasonic extraction propagating through the liquid medium. At the same instant when the bubbles implode, this high frequency creates shock waves and vibrates the plant cell, perhaps rupturing it and releasing its contents. The goal is to accelerate mass movement in order to liberate compound constituents that solvents can quickly extract [40-42].

An analytical balance was used to weigh the *S. polycystum* powder, which was then combined in an Erlenmeyer with 96% ethanol in a ratio of 1:20 before being placed within an ultrasonic apparatus to be extracted for 3x10 minutes with a 5 minute break in each extraction. A thick extract is produced by separating the filtrate and evaporate it with a rotary evaporator. Formula 0 (F0) has no extracts, Formula 1 (F1) contains 3% extract, and Formula 2 (F2) contains 4% extract. These three formulas were created to refine the formulation. One popular gelling ingredient used to make gels is carbomer. This gelling agent has physicochemical characteristics, and the amount of gelling agent utilized influences the gel preparation that is produced.

Organoleptic assay

Shape, consistency, smell, and color of the product are all evaluated during organoleptic testing. Typically, the gel is half-solid in consistency [23].

Drying Time

The dry time test was used to determine how long it takes for a preparation to dry, or how long it takes from when it starts to be applied to the skin until the dry layer was completely dry. Peeling was used to get rid of the preparation from the skin's top layer after it has dried for 15-30 minutes [43]. The three gel mask compositions range in dry time from 15.14 to 17.10 minutes [44].

When compared to masks that contain the *S.polycystum* extract, mask preparations without the extract from *S. polycystum* (F0) dry more quickly. *S. polycystum* extract, which is the active ingredient, impacts the PVA during

the mask's drying phase. Gel masks need to dry for between 15 and 30 minutes before peeling off. Because PVA has sticky characteristics and can form a layer of film that is simple to lick after drying, it helps the mask's peel-off effect [45,46]. PVA acts during the development process by binding together the current water so that water molecules will be close to one another and attract, resulting in greater cohesiveness [31].

The pH Test

The peel-off gel mask was tested for pH, and the results were made to have a pH between 5.66 and 6.04, which is the same as the skin pH. F0 and F1 have a pH of 6, whereas F2 with a pH of 5.66, the skin is feared if the pH of the stock is outside the pH range since it will result in scaly skin and even inflammation. In contrast, if it is above the pH of the skin, it may make the skin feel slick, dry out quickly, and impair the flexibility of the skin [31,47-48].

Homogeneity Test

Homogeneity is a parameter to see whether or not the mixture of the ingredients in the mask formula is evenly effective when applied [9]. From the homogeneity test results of the peel off gel preparation showed that formulas 0, 1, and 2 showed the absence of rough details on transparent glass. It indicates that the preparation made has a homogeneous arrangement. Table 3 presents the characteristics of peel off mask *S.polycystum*.

Antioxidant Activity Test

Based on how antioxidants prevent lipid oxidation, which leads to DPPH free-radical scavenging and, ultimately, free-radical scavenging capability, the DPPH test is used to measure antioxidant activity. Since the analysis just takes a few minutes, the method has been widely employed. The DPPH free radical has a UV-Vis absorbance of 515 nm, is very stable, and interacts with hydrogen-containing compounds. The method depends on antioxidants scavenging DPPH, which, after a reduction process, decolorizes the DPPH methanol solution (Baliyan, 2022). Stable free radicals like DPPH interact with antioxidants by transferring electrons. The DPPH radical changes from purple to yellow when reacts with an antioxidant; converting it to DPPH [20]. The DPPH assay is a quick and easy antioxidant test, and its simplicity and low sample requirement made it the method of choice. UV-Vis spectrophotometry is required for this assay in order to calculate the compound's absorbance [49-51].

Using the free radical capture method DPPH *(1,1-diphenyl-2-picrylhydrazil*) and vitamin C as a control, the antioxidant activity of the *S. polycystum* peel off mask formulation was evaluated. Table 2 presents the measurements of antioxidant activity.

TABLE 2 IC50 Value of peel-off mask

Formulation	IC_{50} Extract (µg/mL)	IC_{50} Vitamin C (µg/mL)
F0	270.03	10.04
F1	129.40	10.04
F2	125.20	10.05

The IC_{50} values of vitamin C were 10 µg/mL and *S. polycystum* peel off mask was 129.40 µg/mL (Table 2). Compared to vitamin C, *S. polycystum* peel off mask formulations have less antioxidant activity. *S. polycystum* peel-off mask has a modest antioxidant activity. A

compound is categorized as a very strong antioxidant if its IC50 value is less than 50 µg/mL, a strong antioxidant if it falls between 50 and 100 µg/mL, a moderate antioxidant between 100 and 150 µg/mL, and a weak antioxidant if its IC50 value ranges from 150 to 200 µg/mL [31].

TABLE 3 Characteristic of peel-off mask S. polycystum

Formula	Shape	Smell	Color	Drying Time (min)	pH	Homogeneity
F0	Gel	Typical	Transparent	15.14	6.00± 0.02	Homogeneous
F1	Gel	Typical	Transparent greenish black	16.05	6.04 ± 0,02	Homogeneous
F2	Gel	Typical	Transparent greenish black	17.10	5.66 ± 0,04	Homogeneous

Conclusion

Based on the results, it is concluded *that S. polycystum* extract can be formulated into a peel-off gel mask. The formula with a concentration of 4% extract has better results, is homogeneous and stable, has a pH of 5.66, a dry time of 17 minutes, and an IC_{50} of 125.20 µg/mL which is a moderate antioxidant.

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Authors' Contributions

All the researchers contributed to this research.

Conflict of Interest

The authors declare that they have no conflict of interest in this study.

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