



Insisiva Dental Journal: Majalah Kedokteran Gigi Insisiva

Website: <http://journal.umy.ac.id/index.php/di/index>

Insisiva
Dental
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Majalah Kedokteran Gigi Insisiva

Research Article

Formulation and Physical Characteristics Evaluation of *Sargassum* sp. Lip Gel for the Exfoliative Cheilitis Treatment

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DOI: 10.18196/di.v14i1.23659

Abstract

Exfoliative cheilitis is a disease that often occurs, especially in Indonesia, exacerbated by hot weather and vehicle exhaust pollution, which can cause dry and cracked lips. The components found in *Sargassum* sp. are beneficial in enhancing the effectiveness of vitamin C, aiding skin regeneration, and potentially acting as antioxidants to combat free radicals. To develop a lip care formulation from *Sargassum* sp. seaweed to overcome the problem of exfoliative cheilitis, *Sargassum* sp. was crushed and extracted to produce three kinds of formulations that tested for homogeneity using methylene blue, which was dripped on each replication on a watch cup and stirred until a homogeneous mixture between the oil and water content. Viscosity was tested using a Brookfield viscometer, the organoleptic assessment was done visually, the pH test was performed using a pH meter, occlusivity was tested using filter paper, the stability test was evaluated using a centrifuge, and the adhesion test was conducted using an adhesion test tool. The study results revealed in the organoleptic test that the form of the three formulations was the gel form, with transparent yellowish to yellowish brown color. At the same time, the aroma was slightly pungent, which is typical of *Sargassum* sp. (F1-2). In the viscosity test, there were significant differences between preparations F1(48.740±408.412), F2(9.420±274.955), and F3(2200±91.652). In the pH test, there were significant differences between F1(5.657±0.019), F2(6.838±0.058), and F3(8.960±0.112). All formulations were homogeneous and stable after centrifugation for 30 minutes in the homogeneity and stability tests. In the occlusivity test, there was no significant difference in preparations F1-3, and only significant differences were found in the occlusivity test at 24 and 48 hours in the F3 formulation. In the adhesion test, there were significant differences between F1(2.4367±0.168), F2(2.1467±0.127), and F3(1.3467±0.145). Formulations 1 and 2 met the gel preparation test criteria, while formulation 3 did not meet the gel preparation test criteria.

Keywords: formulation; physical characteristics; lip gel; *Sargassum* sp.; exfoliative cheilitis

INTRODUCTION

Exfoliative cheilitis is one of the common diseases in Indonesia. Hot weather conditions and vehicle pollution can cause lips to dry and chapped. Exfoliative cheilitis can also be caused by daily activities such as excessive lip licking. This is caused by saliva, which damages the thin lip skin layer and takes its natural moisture. Exfoliative cheilitis are very resistant to various types of treatment, making it very difficult to

determine which therapy provides optimal results.¹

Sargassum sp. is one of the seaweeds that contains iodine and secondary metabolites, such as flavonoids, alkaloids, saponins, phenols, and triterpenoids, which can function as anti-cholesterol, biofuel, biofertilizer, antibacterial, antitumor, anticancer, antifouling, antiviral, antifungal, and cosmetic cream.²

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The contents contained in *Sargassum sp.* are beneficial in enhancing the effectiveness of vitamin C, which can help the skin regeneration processes and has the potential as an antioxidant that can suppress free radicals.³ The use of *Sargassum sp.* as a lip gel ingredient can also provide additional attraction for consumers who care about the environment and seek products made from natural and environmentally friendly materials.

MATERIALS AND METHODS

This experimental laboratory research used *Sargassum sp.* as a test material. This laboratory study was conducted to see the characteristics of the gel preparation formulation with differences in the concentration of the test material by 5%, 15%, and 25%. Each test material variable was repeated three times, and seven characteristic tests included organoleptic, viscosity, pH, homogeneity, occlusivity, stability, and adhesion. This study was carried out after obtaining approval from the Ethics Commission of the Nala Husada Dental and Oral Teaching Hospital Surabaya, EC/015/KEPK.RSGMNH/IX/2024.

Sargassum sp. Extraction Method

The production of ethanol extract of 96% *Sargassum sp.* used the maceration method. A total of 100 grams of *Sargassum sp.* powder was placed in a container and then given 96% ethanol with a 1:10 ratio with *Sargassum sp.*, which had been submerged in 96% ethanol and left for 72 hours in a sheltered place while repeatedly mixed. Afterward, it was filtered and macerated two times. Next, all the filtrate was collected and attached using the Rotary Evaporator to

A variety of forms of preparation are in great demand and are now prevalent among women, especially teenagers, and they have various formulations, including lip gloss, lip cream, lip tint, lip gel, and lip balm. Lip gel has a very good effect on lip care problems; it has properties that are almost similar to lip balm, protecting lips from environmental factors and providing maximum moisture for healthy lips.

separate the ethanol from the liquid extract into thickness.^{4,5}

Process of Making Lip Care Preparations

The process of making lip care with gel preparations from *Sargassum sp.* began by washing the *Sargassum sp.* with running water until it was clean of dirt. This process was followed by drying using the freeze-dry method for two days. The next step was to grind the dry results of the *Sargassum sp.* blender for the extraction process using ethanol for three days.

All additional ingredients for making lip care preparations, such as carbomer 940, nipagin, glycerin, triethanolamine (TEA), tween, and distilled water, were weighed and mixed with sargassum extract, which was done on a hot plate so that all the ingredients could be combined and then let sit at room temperature.⁶ Three formulation ingredients were created, and five gel preparation characteristic tests were conducted: viscosity test, pH test, occlusivity test, stability test, and adhesion test. Each test was repeated three times, and the data were tabulated and analyzed.

Table 1. Lip Care Formulations

Materials	FI	FII	FIII
<i>Sargassum</i> sp. *	5%	15%	25%
Carbomer 940	1 gram	1 gram	1 gram
Nipagin	0.2 gram	0.2 gram	0.2 gram
Glycerin	0.1 gram	0.1 gram	0.1 gram
TEA	0.5 gram	0.5 gram	0.5 gram
Tween	10%	10%	10%
Distilled water	100 mL	100 mL	100 mL

* Concentration difference

Characteristic Tests

Organoleptic Test

The panelists in this test consisted of ten people and were asked written questions to be answered. The questions asked were regarding observations of the *Sargassum* sp. preparation formulation's shape, color, and taste. Color and shape examinations were carried out visually by examining a clean container from the outside under good lighting, blocked from reflections in the eyes, and a black and white background, with a series of contents running with a rotating action, which must be completely free of small particles visible. Identification of odor characteristics could be done by smelling the sample directly.⁷

Viscosity Test

The first preparation was the Brookfield viscometer, which was set using spindle number seven. Next, one hundred milliliters of each *Sargassum* sp. extract gel formulation was taken, and each formulation was tested three times as a replication. After that, the spindle was set on the viscometer, and the rotation speed was set at 10 RPM. The spindle was then dipped into the gel preparation, and the viscometer lever was slowly lowered until the measurement position was ready. The spindle motor was turned on, and it rotated for approximately 30 seconds to 1 minute until the gel preparation was stable. Once stable, the monitor on the viscometer would

display the viscosity value of the gel preparation being tested. The value was then recorded as a measurement result.⁸

pH Test

This test utilized a digital pH meter to measure the acidity or alkalinity of the *Sargassum* sp. extract gel preparation. The process began by taking one hundred milliliters of each gel formulation and dividing it into three replications. This was done to ensure the accuracy and consistency of the measurement results. Measurements were made by dipping the pH meter electrode into the gel preparation placed in a beaker. The electrode was left in the gel until the pH number displayed on the screen was stable. Afterward, the pH number was recorded as the measurement result. This procedure was repeated three times for each replication of each formulation tested.

Homogeneity Test

Measurement of homogeneity of *Sargassum* sp. extract gel preparation was done using methylene blue, which was dripped into three replications of formulations 1, 2, and 3 on a watch cup. It was stirred until the oil and water content looked homogeneous. This test examined whether the lip preparation was homogeneous or if there were still coarse grains in the preparation characteristic test.¹⁰

Occlusivity Test

Occlusivity testing is a test performed to see the absorption capacity of heat temperatures. This test was conducted using filter paper, which was left for 1 x 24 hours. Previously, it was dripped with IPM until alkaline was reached, then covered with aluminum foil. After that, the nine-bottle vial (3xreplications) was filled with 10 ml of distilled water solution, weighed first, covered with filter paper, and then coated with parafilm. A sample of 0.5 grams was taken and smeared on the filter paper. Then, it was placed in an oven at a temperature of 40°C.¹¹

Stability Test

The measurement of the stability test using centrifugation was replicated three times for each formulation. Each replication of the three formulations was

put into a tube of 90 ml. Then, each replication of the first formulation was put into the device to be tested for centrifugation for 30 minutes. After centrifugation was done, each replication tube of Formula 1 was removed from the device. Do the same for the second and third formulations.¹²

Adhesion Test

The adhesion test was performed to determine how long it took for the lip gel to stick to the lips when applied. It was carried out with 1 gram of gel preparation placed between two object glasses on the adhesion tester, then a load of 50 grams for 1 minute. The load was lifted, the 50-gram load was put on the lip, and the time of gel lubrication was recorded. The ideal adhesion test of lip gel formulation was around 2-4 seconds.¹³

RESULTS

Organoleptic Test

The results of the organoleptic test on texture demonstrated that all ten respondents agreed that the manufacture of gel formulation from *Sargassum sp.* had obtained a semisolid gel form. The results of the organoleptic test on color showed that F1, FII, and FIII had different colors. Respondents considered F1 to be

transparent and yellowish, F2 to be transparent and brownish, and F3 to be yellowish and brownish. This can be generalized that the higher the concentration, the more concentrated the color will be. The results of the organoleptic test on aroma revealed that the F1 and F2 formulations had a slightly pungent *Sargassum sp.* typical aroma, while F3 had a more pungent *Sargassum sp.* typical aroma.

Table 2. Organoleptic Test Result

Preparation	Texture	Color	Aroma
F1	Gel	Transparent yellowish	Slightly pungent aroma <i>Sargassum sp.</i> typical
F2	Gel	Brownish- yellow	Slightly pungent aroma <i>Sargassum sp.</i> typical
F3	Gel	Yellowish Brown	Pungent aroma <i>Sargassum sp.</i> typical

Viscosity Test

In the viscosity test, F3 had an ideal viscosity (2,200 ± 43.577 cPs), in accordance with the optimal range, while F2 and F3 had higher viscosities, which

could affect the stability and spreadability of the gel preparation.

Table 3. Viscosity Test Result

Preparation	Viscosity (cPs)
F1	48.740 ± 408.412 ^a
F2	9.420 ± 274.955 ^b
F3	2.200 ± 91.652 ^c
p-value	0.00

Note: a,b,c Difference between the groups with a significance level of 5% ($p < 0.05$).



Figure 1. Lip Gel Viscosity Test

In the ANOVA and Tukey-HSD statistical tests, significant differences were found between the formulations (F1-3).

pH Test

The pH test aimed to determine the acidity level of the lip care gel preparation and to ensure that the preparation did not irritate

the lips. The pH moisturizer preparation value of the lip must meet the requirements of the Indonesian Nation Standard SNI 16-4769-1998, namely the pH lip of moisture, which is in a range of 6.5-7.9. In this study, the ideal pH of lip care gel was found in the F2 formulations.

Table 4. pH Test Result

Preparation	pH
F1	5.658 ± 0.019 ^a
F2	6.838 ± 0.058 ^b
F3	8.960 ± 0.112 ^c
p-value	0.00

Note: a,b,c Difference between the groups with a significance level of 5% ($p < 0.05$).

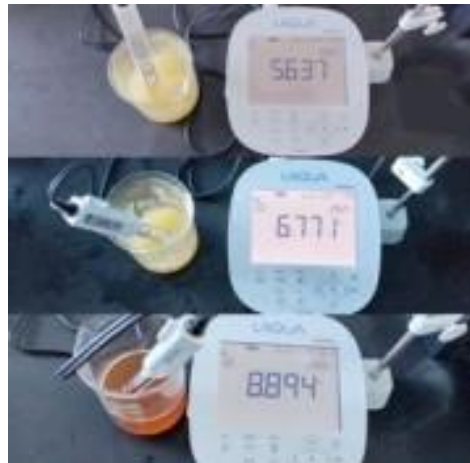


Figure 2. Lip Gel pH Test

The results of Kruskal-Wallis and Mann-Whitney tests exhibited significant differences between formulations (F1-3).

Homogeneity test

Homogeneity tests on F1, FII, and FIII

uncovered homogeneous results indicated by all particles in the observation in the watch cup being evenly dispersed and showing no coarse particles in each formula and no gel particles separating between oil and water in all samples tested.

Table 5. Homogeneity Test Result

Preparation	Homogeneity test
F1	Homogeneous
F2	Homogeneous
F3	Homogeneous

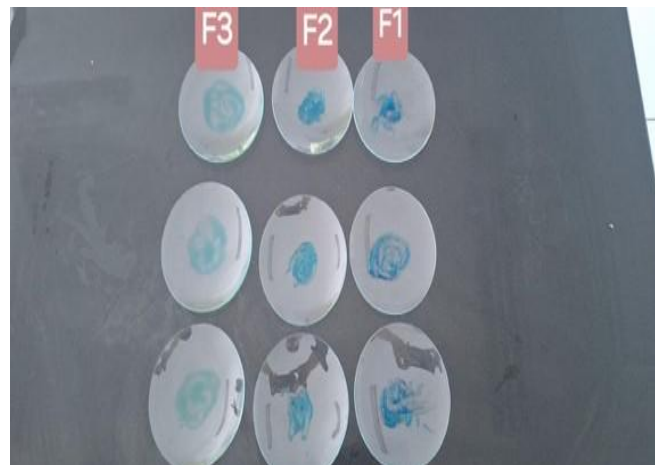


Figure 3. Homogeneity Test of Lip Care Preparations

Occlusivity Test

The results of the occlusivity test showed no significant difference at 24 in each formulation (F1-3), as well as in the first

48 hours. In the difference test between 24 and 48 hours in each formulation, there was a significant difference in F3.

Table 6. Occlusivity Test Result

Preparation	Occlusivity-24	Occlusivity-48	p-value
F1	4.90%±0.636%	7.37%±0.092%	0.281***
F2	0.41%±0.036%	7.37%±0.083%	0.109****
F3	0.96%±0.080%	2.95%±0.088%	0.001***
p-value	0.338*	0.957**	

Note: *ANOVA; **Kruskal Wallis; ***paired t-test; ****Wilcoxon

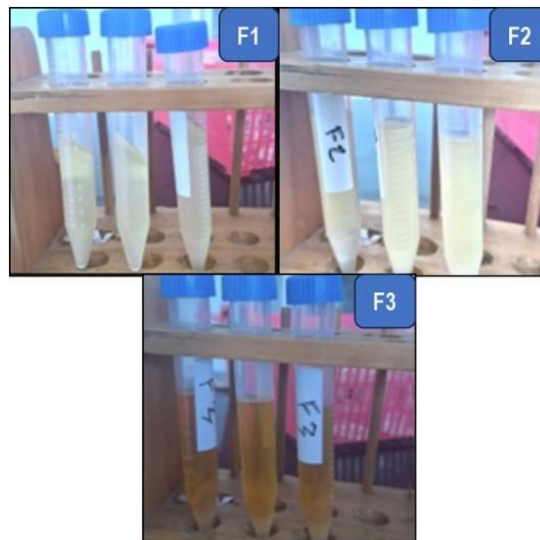
Stability Test

Based on the stability test of gel preparation containing *Sargassum* sp. extract, which was centrifuged for 30 minutes, there was no change in color, odor, shape, or

homogeneity in the preparation. This denotes that the gel preparation containing *Sargassum* sp. was stable, indicated by the absence of separate oil and water phases.

Table 7. Stability Test Result

Preparation	Stability test
F1	Stable
F2	Stable
F3	Stable

**Figure 4.** Lip Gel Stability Test

Adhesion Test

Data analysis using the One-Way ANOVA test showed that the significance value for testing the adhesive power on F1, F2, and F3 was $p < 0.05$, meaning that there was a significant difference in each formula.

Then, it was continued with the Tukey HSD method, which obtained the results of a significant difference ($p < 0.05$) between F3 with F1 and F2. The results for F1 and F2 revealed no significant difference ($p > 0.05$).

Table 8. Adhesion Test Result

Preparation	Adhesion test
F1	2.437±0.168 ^a
F2	2.147±0.127 ^a
F3	1.347±0.145 ^b
p-value	0.000

Note: a,b Difference between the groups with a significance level of 5% ($p < 0.05$).

DISCUSSION

In organoleptic observations, the shape of the gel preparations in F1, F2, and F3 was formed well. Although there were differences in concentration, the shape of the gel did not change. This indicates that the formulation is correct when making gel preparations. This formulation used carbomer 940, which is a high molecular weight acrylic acid polymer gelling agent that is widely used in the pharmaceutical industry.¹⁴

This differs from organoleptic observations of color, where the higher the concentration, the more concentrated the brownish color that appears accompanied by a strong aroma typical of *Sargassum sp.*¹⁵ Therefore, in order to be accepted by users, further evaluation is needed so that the color and aroma can be attractive and make the preparation fresher in aroma by adding corrigent odors and colors.¹⁶

The optimal viscosity for a topical gel lies within a range that ensures ease of application, effective spreadability, and sufficient drug release. Although the precise range can differ based on the formulation and intended use, a commonly accepted guideline is between 2,000 and 4,000 centipoise (cP) or 2–4 Pa.s. If the viscosity is too high, the gel may be hard to apply and spread, whereas too low a viscosity could result in an overly runny gel.¹⁷ The results of the study showed that F3 met the ideal gel viscosity criteria, where statistical tests exhibited significant differences between F1, F2, and F3. Formulations of F1 and F2 can be improved by reducing carbomer 940 because the carbomer content as a gelling agent allows a minimum of 0.5%.¹⁴

The results of pH testing on three gel

formulations revealed that formulation F2 had the most suitable pH, which was within the physiological pH range of the oral cavity of 6.5–7.9.^{8,18} In contrast, formulation F1 showed a slightly more acidic pH value, and F3 showed a more alkaline condition. Statistical analysis using the Kruskal-Wallis test demonstrated a statistically significant difference between the three formulations, which was most likely influenced by the difference in concentration of the active ingredients used.¹⁹

Research conducted by Wahyudi et al.²⁰ emphasized that pH testing on preparations applied to the oral cavity aims to assess the pH balance of the preparation with the physiological conditions of the oral environment. Too acidic pH can cause irritation to the mucosa, increase the growth of acidogenic bacteria, and increase the risk of tooth enamel demineralization. Therefore, the preparation used should have a pH close to the normal pH value of the oral cavity. In addition, spreadability testing is carried out to determine the ability of semisolid preparations to spread optimally without requiring excessive pressure, thus allowing for comfortable and painless application to the user.²¹

Additionally, the occlusivity or hydration test is used to assess the ability of a preparation to prevent evaporation. The lower the distilled water that evaporates at a temperature of 40°C, the better the preparation is in preventing evaporation. Preparations that do not evaporate easily can maintain lip moisture for longer. This study showed that F1 and F2 had the same occlusivity, so this formulation could maintain lip moisture well. In comparison, F3 had a fairly drastic change from the first

24 hours to 48 hours, which allows for accelerated evaporation of the material. However, this lip gel did not allow for a day or even two days in a row, so it can be said that this formulation met the requirements in terms of maintaining moisture.²²

The stability test in this study aimed to see the stability between the oil and air phase, where the study results showed that the gel was stable, and no separate preparations were seen. This indicates that no or little oil element was contained in *Sargassum* sp. This is supported by the research of Gazali et al.²³, who took *Sargassum* sp. from the west coast of Aceh. The study showed that phenolic content is the most significant component of *Sargassum* sp., so in the formulation of the gel preparation tested for centrifugation at a speed of 3000 rpm for 30 minutes, it remained homogeneous and stable.

Furthermore, the adhesion test aimed to evaluate the extent to which the lip balm could adhere to the epidermis of the lips. The longer the product adheres, the better

CONCLUSION

The formulation of lip gel from *Sargassum* sp. needs to consider the characteristics of biomaterials that can support the effectiveness of exfoliative cheilitis treatment. From the results of the characteristic test of three gel formulations, it may be concluded that the 5% concentration lip gel displays superiority in organoleptic and adhesion. In addition, the 15% gel formulation is safe in terms of dryness and prevents lip irritation, and the 25% gel formulation is superior in viscosity

the quality of the adhesion because users do not need to reapply the lip balm repeatedly. The minimum acceptable adhesion criteria are not less than 4 seconds.²⁴ Based on the data in Table 8, all the formulas tested have not met these standards, where the F3 formulation had the lowest adhesion and was significantly different compared to F1 and F2. Therefore, reformulation efforts are needed to increase the adhesion of the lip gel. For the use of ingredients with strong layer-forming properties, it is necessary to add polymers or waxes that create strong bonds with the lips. In particular, ingredients such as polybutene, certain waxes, and oil-gelling agents can help achieve better adhesion.²⁵ Increasing the concentration of *Sargassum* sp. has been shown to reduce the adhesion of lip gel. This is because the characteristics of the active content of *Sargassum* sp., which is polar, are unable to increase the adhesion ability of the lip gel preparation (Gazali, 2018).

and occlusivity. This formulation necessitates enhancement, particularly in the event that in vivo testing is implemented to enhance the real effectiveness of the product in experimental animals.

ACKNOWLEDGEMENT

This research was supported by the Grants-in-Aid for Student Creativity Program-Exact Research from the Indonesian Ministry of Research and Technology.

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