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Formulation of Vitamin C Serum Gel Using Sodium Alginate from Brown Algae (*Sargassum polycystum*) as Gelling Agent

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ABSTRACT

Background: Alginate contained in brown algae can be used as a gelling agent on cosmetics product. In this study, alginates from brown algae is formulated on antioxidant serum gel. Vitamin C was used as a model of active ingredient since its known as potent antioxidants. In order to obtain a serum gel preparation with good physical characteristics, optimization was also carried out in the formula between sodium alginate as a gelling agent and propylene glycol as a humectant.

Aim: The purpose of the research is to determine the optimum ratio of sodium alginate to propylene glycol to provide good physical characteristics of vitamin C serum gel.

Method: The extraction method used to obtain sodium alginate from brown algae (*Sargassum polycystum*) using the acid pathway extraction method. The method used to optimize sodium alginate and propylene glycol is the Simplex Lattice Design method with Design Expert software version 10.0.1. Optimization parameters included tests of viscosity, spreadability, adhesion, and pH. Penetration test is conducted using Franz diffusion cells, and the stability test of the dosage form is conducted using the cycling test method.

Result: The yield of sodium alginate extraction was 20.61%. Results of analysis software Design Expert version 10.0.1 show that sodium alginate could increase viscosity, adhesion, and pH of serum gel, and reduce the spreadability of vitamin C serum gel, while propylene glycol could increase all the responses.

Conclusion: The yield of brown algae (*Sargassum polycystum*) obtained was 20.61% with the optimum ratio of sodium alginate to propylene glycol in the preparation of serum gel vitamin C being 7.56% : 10.44%.

Keywords: Brown algae, optimization, propylene glycol, sodium alginate, serum gel

INTRODUCTION

Algae is one of the abundant natural resources in Indonesian waters. Based on the pigment content, algae is divided into three, namely red algae (Rhodophyta), green algae (Chlorophyta), and brown algae (Phaeophyta) (Maharani et al., 2017). *Sargassum polycystum* is a type of brown algae that is commonly found in Indonesian waters. Algae cell walls contain alginic acid, laminarin, and fucoidan. The most abundant chemical compound in brown algae is alginate (Yunizal, 2004).

On Pailus Beach, Jepara, Central Java, brown algae (Sargassum polycystum) is often found on the beach because it is carried by the waves. However, the local community has not yet used the brown algae, so the brown algae (Sargassum polycystum) only becomes waste on the beach. Brown algae has a high alginate content so it can be used as a gelling agent. Therefore, the authors intend to utilize alginate from brown algae (*Sargassum polycystum*) as a gelling agent in cosmetic preparation of serum gel.

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There are two methods for extracting sodium alginate from brown algae, namely the acid pathway method and the calcium pathway method. In this study, the acid path extraction method was used. This method was chosen because the process is shorter than the calcium pathway extraction method, so it can shorten the research time.

The alginate then was formulated into a serum gel preparation to determine the ability of the alginate to form a gel. The serum dosage form was chosen because serum is one of the cosmetic preparation innovations which is very popular currently. It contains high concentration of active ingredients so they have a more potent effect than other cosmetic preparations. According to Draelos (2010), cosmetic serum is a preparation that contains more bioactive ingredients and less solvent.

In order to obtain a serum gel preparation with good physical characteristics, the formula was optimized between sodium alginate as a gelling agent and propylene glycol as a humectant. Both of these materials are optimized because they affect the physical characteristics of the serum gel preparation. The ability of sodium alginate to form a gel will affect the consistency of the preparation. Whereas propylene glycol as a humectant has the property of preventing syneresis, namely the release of water from the gel structure, so as to prevent a decrease in gel viscosity.

Today's cosmetics have offered various solutions to prevent signs of aging and wrinkling of the skin. It is known that vitamin C is one of the most potent antioxidants that can prevent signs of aging. Therefore, vitamin C was chosen as model of the active ingredient in gel serum preparations (Colven and Pinnell, 1996). In the serum gel preparation, 10% vitamin C is added. This concentration was chosen because 10% is the concentration commonly used in vitamin C serum preparations that are sold in the market. The range of topical content that can be used as a cosmetic preparation is from 3% to 17% (Bisset et al., 1990).

METHODS

Sodium Alginate Extraction

Sodium alginate which is a gelling agent from the preparation, was obtained through alginate extraction from brown algae (*Sargassum polycystum*). The extraction of sodium alginate uses the acid path method. First of all, the brown algae was cleaned from adhering dirt and cut into smaller pieces, washed and dried in the sun for 2-3 days. Then, it was soaked using 1% $CaCl_2$ for 30 minutes and washed. The second soak was done with 5% HCl for 30 minutes and washed. After that, 0.5% KOH solution was added for 60 minutes at 50-60°C and washed. Extraction was carried out with 2.25% Na_2CO_3 at 50-60°C for 1 hour and filtered. The filtrate obtained was blanched with 10% NaOCl, stirred and left for 5 hours. Precipitation with 5% HCl was carried out until a precipitate of alginic acid was formed, washed and filtered. After that it was neutralized with 10% Na_2CO3 at pH 6-7, stirred and filtered. Purification was carried out by 95% isopropanol and dried at 50-60°C for 17 hours, then milled to obtain sodium alginate in powder form.

Evaluation of Extracted Sodium Alginate

The sodium alginate is then subjected to several tests to determine its quality, including :

1. Yield

The yield of sodium alginate extracted from brown algae was calculated by calculating the weight percentage of extracted sodium alginate compared to the weight of brown algae samples used.

2. Ash Content

Analysis of ash content was carried out according to the AOAC method (1990), by burning the sample in a muffle at 600°C for 3 hours. The alginate ash content is expressed by calculating percentage of ash residue

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after the process compared to initial weight of sample.

3. Water content

Moisture analysis was carried out according to the gravimetric method by drying the alginate sample at 105°C for 30 minutes until a constant weight was obtained (AOAC, 1990). The water content of sodium alginate is expressed by calculating the dry weight percentage compared to the initial weight. The requirement for sodium alginate water content is less than 15% (Winarno, 1996).

4. Functional Group Analysis

Functional group analysis of extracted sodium alginate was carried out using the FT-IR instrument (Agilent Technologies Cary 630 FTIR). The extracted sodium alginate samples were analyzed for their functional groups based on the spectral results that emerged from the FT-IR and compared with the spectra of sodium alginate (Sigma 180947).

Formulation of Sodium Alginate in Serum Gel Preparations

In order to obtain serum gel preparations with good physical characteristics, in this study optimization was carried out between sodium alginate extracted from brown algae (*Sargassum polycystum*) and propylene glycol. The design formula for vitamin C serum gel preparation with sodium alginate extracted from brown algae (*Sargassum polycystum*) as a gelling agent was created using the design expert program version 10.0.1 is shown in Table 1.

Table 1. Formula Design for Serum Gel Vitamin C

Formula	**F1 (%)**	**F2 (%)**	**F3 (%)**	**F4 (%)**	**F5 (%)**	**F6 (%)**	**F7 (%)**	**F8 (%)**	**F9 (%)**	**F10 (%)**
Vitamin C	10	10	10	10	10	10	10	10	10	10
S-Alginate	5,5	3	6,75	4,25	5,5	8	8	3	8	3
Propylenglicol	12,5	15	11,25	13,75	12,5	10	10	15	10	15
S-Metabisulfite	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5
Methyl Parabene	0,1	0,1	0,1	0,1	0,1	0,1	0,1	0,1	0,1	0,1
TEA	0,74	0,74	0,74	0,74	0,74	0,74	0,74	0,74	0,74	0,74
Aquadest	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100

The gel was prepared by sprinkling sodium alginate into a cup containing hot distilled water (Mixture 1). Furthermore, methyl paraben is dissolved in propylene glycol because methyl paraben has a higher solubility in propylene glycol compared to water. The active ingredients vitamin C and sodium metabisulfite are dissolved in distilled water and mixed into a mixture of propylene glycol and methyl paraben (mixture 2). After the sodium alginate from mixture 1 expands and reach room temperature, mixture 1 and mixture 2 are combined and stirred until homogeneous. The coloring agent and fragrance are added to improve the appearance of the preparation. The preparations are then put into the container.

Evaluation of Dosage Form

To determine the optimal formula, the response of viscosity, spreadability, adhesion and pH was used to obtain a combination of propylene glycol and sodium alginate that produced the best physical characteristics. Furthermore, preparations made based on the optimal formula obtained are evaluated, which include :

1. Organoleptic

Organoleptic examination was conducted by observing consistency, color, and odor of antiaging gel serum preparations from brown algae alginate (*Sargassum polycystum*).

2. Homogeneity

This examination was carried out by applying serum gel alginate extract of brown algae (Sargassum polycystum) to a material with a smooth surface and observing the homogeneity of the color dispersion formed

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(Siregar and Utami, 2014).

3. pH

Brown algae (*Sargassum polycystum*) serum gel samples is tested with a calibrated pH meter (Hanna Instrumen pH 210). The cathode of the pH meter is immersed in the preparation and the pH of the preparation produced during the test is recorded (Siregar and Utami, 2014).

4. Viscosity

Gel serum preparations were measured directly using a Brookfield viscometer (Brookfield DV-1 Prime). Viscosity is seen on the scale was read after stability is achieved (Martin, 2008).

5. Spreadability

As much as 0.5 g of serum gel preparation is placed in the middle of the spreadability test equipment. The top glass is weighed, then placed on top of the gel serum and left for one minute. The diameter of the distribution of the gel serum formed was measured by measuring average diameter from several sides. An additional load weighing 50 grams was placed on the glass, it is allowed to stand for 1 minute and the diameter of the spread of the serum gel preparation was recorded. The experiment was continued each time with the addition of 50 grams of weight and the diameter of the spread of serum gel was recorded for 1 minute until a constant spreadability was obtained (Astuti et al., 2010).

6. Adhesion tests

As much as 0.25 gram of serum gel preparation is placed between 2 glass objects that have been determined by the area, then pressed with a load of 1 kg for 5 minutes. The object glass was installed on the test equipment, the load weighing 80 grams was released and the time was recorded until the two object glasses were released (Astuti et al., 2010).

7. Preparation Penetration Test for Optimum Formula

Penetration test of the preparation was carried out using the Franz diffusion (Preme Gear) cell. In this test, a cellophane membrane was used which is analogous to human skin. The cellophane membrane was hydrated in phosphate buffer pH 7.4 at 37°C to mimic human physiological conditions. The penetration test with the Franz diffusion method was carried out for 5 hours by taking samples at 10, 30, 60, 90, 120, 180, and 240 minutes. Next, the absorbance of each sample was measured using a UV spectrophotometer (Shimadzu 1240), and the concentration was calculated.

8. Stability Test

The stability test of the preparation was carried out using the cycling test method. The cycling test was carried out in 5 cycles, each cycle consisting of storage for 24 hours at a cold temperature of 4°C and 24 hours at 40°C. After 5 cycles of the cycling test, the characteristics of the serum gel gel serum were tested including pH, viscosity, spreadability, and adhesion tests.

RESULTS AND DISCUSSION

Extraction results of sodium alginate from brown algae are presented in Table 2. Extracted sodium alginate has % yield, % ash content and % water content which meet the quality standard requirements (Food Chemical Codex, 1981).

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Table 2. Quality of Alginate from Sargassum polycystum

Testing	Result	Standard Requirements
% yield	20.61	>18%
% ash content	20.68	18-27%
% water content	8.54	<15%

Comparison of the spectra of standard sodium alginate with extracted sodium alginate in (Figure 1) showed that sodium alginate extracted from brown algae (*Sargassum polycystum*) has a spectrum that is almost the same as standard sodium alginate. Standard of sodium alginate has a sharper absorption intensity. The intensity of absorption is affected by the number of alkyl groups attached, in which the more alkyls attached the less intensity of absorption. The extracted sodium alginate has a lower absorption intensity compared to the standard sodium alginate sold in the market because the extracted sodium alginate still contains impurities and has a lower purity level than the standard sodium alginate.

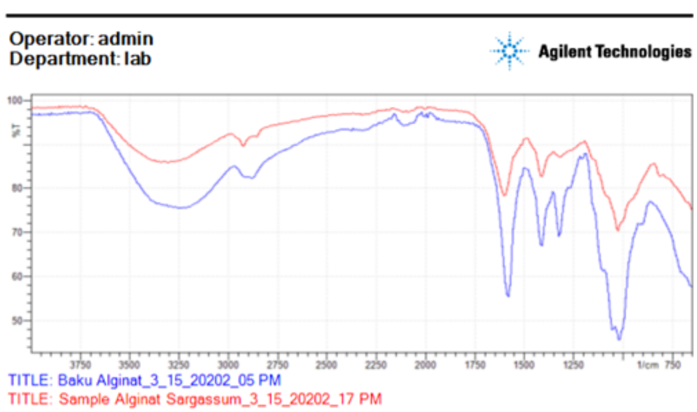

Figure 1. Comparison of the Spectrum of Sodium Alginate Extracted from Sargassum polycystum with Comparative Sodium Alginate (Sigma 180947)

Evaluation of physical characteristics of the serum gel preparation included the viscosity test, spreadability test, adhesion test, and pH. These parameters were selected to determine the optimum formula for vitamin C serum gel preparation. The test was conducted to determine how the effect of sodium alginate and propylene glycol concentrations and their interactions on the physical characteristics of vitamin C serum gel preparations. The results of characteristic test for vitamin C serum gel preparations with alginate extracted from brown algae for each formula is presented in Table 3.

Table 3. Physical Characteristics Test Results of Vitamin C Serum Gel

Testing	Formula

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	I	II	III	IV	V	VI	VII	VIII	IX	X
Viscosity (cPs)	628	498	843.8	575.9	627.9	1314	1315	337	1314	498
Spreadability (cm)	8.28	12.45	9.45	11.83	9.83	7.65	6.58	11.73	7.23	12.9
Adhesion Test (second)	1.38	1.25	1.54	1.28	1.34	1.8	1.8	1.29	1.78	1.31
pH	3.15	3.12	3.18	3.12	3.14	3.26	3.27	3.12	3.28	3.12

Determination of the optimum formula was determined by Design Expert 10.0.1 in accordance with the criteria and weights of the optimization parameters that have been determined. The formula predicted by Design Expert 10.0.1 as the optimum formula was a formula with a composition ratio of 7.56% sodium alginate and 10.44% propylene glycol with a desirability value of 0.851.

The evaluation parameter to test the optimum formula for serum gel preparations (Figure 2) are including tests of viscosity, pH, spreadability, adhesion. The predicted results of the Simplex Lattice Design equation with the research results including pH, viscosity, spreadability and adhesion of serum gel preparations were validated with a T-test (Table 4).

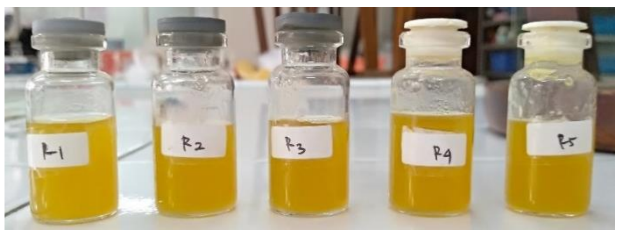

Figure 2. Vitamin C Serum Gel Preparation with Optimum Formula.

Table 4. Different Test of Optimum Formula Theoretical Results of Expert Design with Experimental Results

Response Parameters	Experiment Results	Predicted Results	Sig (2-tailed)	Conclusion
Viscosity (cPs)	1153,6	1150,002	0,071	Not significantly different
pH	3,224	3,238	0,052	Not significantly different
Spreadability (cm)	7,62	7,665	0,483	Not significantly different
Adhesion Test (second)	1,688	1,692	0,345	Not significantly different

Table 4 shows a comparison between the experimental results of each test parameter with the theoretical results for validating the Simplex Lattice Design equation. The optimum formula shows insignificantly different results for each test parameter, which can be seen from the value of sig(2-tailed) > 0.25 which indicates that the experimental results are not significantly different from the theoretical results from the Design Expert.

In this study, an *in vitro* penetration test was also carried out using a Franz diffusion cell. Tests are conducted to determine the amount of vitamin C that can penetrate through the skin over a certain time interval. The average cumulative amount of vitamin C that penetrates each sampling time through the cellophane membrane from each replica of the vitamin C serum gel preparation can be seen in the graph in Figure 3.

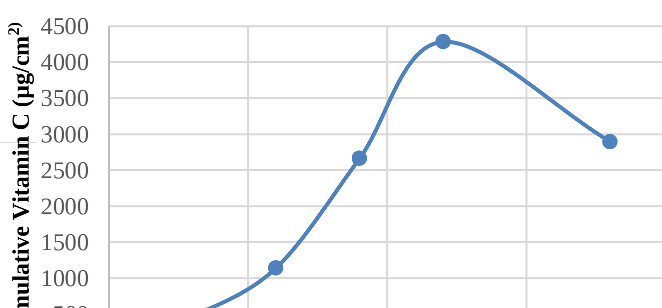

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Figure 2. Graph of Cumulative Penetration of Vitamin C (µg/cm2)

From the results of research conducted, the rate of penetration has increased from the first minute to reach the peak, and then decreased. This can be seen in the levels of vitamin C which increased in the first few minutes and then decreased when entering the 180^{th} minute. The decrease in levels was due to the smaller concentration gradient so that the penetration rate decreased. Vitamin C in serum gel preparations penetrates into the skin in a relatively short time so that at 180 minutes and above, the penetration process of the active substance no longer occurs (Martin and Cammarata, 2008).

The results of the cycling test of vitamin C serum gel preparations for 5 cycles did not show significant changes in the physical characteristics of the serum preparations. Organoleptics of vitamin C serum gel preparation before the cycling test and after the cycling test did not show any change in color, odor, or consistency of the preparation.

Table 5. Physical Characteristics of Serum Gel Preparations
Before and After the Cycling Test

Testing	**Before *cycling test* (x ± SD)**	**After *cycling test* (x ± SD)**
Viscosity (cPs)	1153.6 ± 3.28	1154 ± 3.61
pH	3.224 ± 0.01	3.218 ± 0.02
Spreadability (cm)	7.62 ± 0.1304	7.58 ± 0.08
Adhesion Test (second)	1.688 ± 0.0083	1.69 ± 0.01

CONCLUSION

The yield of brown algae (*Sargassum polycystum*) obtained was 20.61% with the combined composition with propylene glycol in the preparation of serum gel vitamin C being 7.56% : 10.44%. It is necessary to continue the chemical stability test on the optimum formula for vitamin C serum gel preparation to determine whether there is a change in vitamin C levels in the preparation by assaying using UV spectrophotometry.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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