Optimization of temperature and time in carrageenan extraction of seaweed (*Kappaphycus alvarezii*) using ultrasonic wave extraction methods

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Abstract. Carrageenan is a hydrocolloid compound from *Rhodophyceae* extraction of *K. alvarezii* using alkali solvents. Conventional extraction methods that have been using certain solvents in large volumes require a long time and low yields resulting in high production costs and large amounts of waste which pollute the environment. These adverse effects can be minimized by extraction with the help of ultrasonic waves which will provide an intensive stirring effect to damage the cell walls of seaweed so that the process of mass transfer will be faster. The extraction temperature greatly influences the quantity and quality of carrageenan which is often carried out in conventional extraction. In carrageenan extraction activities from *K. alvarezii* with the help of ultrasonic waves, the effects of temperature and time on these two parameters will also be studied. This activity was conducted to determine the time and temperature of carrageenan extraction from *K. alvarezii* seaweed using an ultrasonic frequency of 40 kHz with temperature variations of 30, 40, 50, 60, and 70 °C, and time variations of 20, 25, 30, 35 and 40 minutes. The results of extraction of *K. alvarezii* seaweed with a time variation of 40 minutes and temperature variation is 70 °C, with the optimum value of temperature and extraction time is 70 °C for 30 minutes which produces carrageenan as much as 44.46%. The carrageenan quality test produced water content ranging from 6.27-14.18%, ash content 12.33-13.69%, viscosity of 10.4-27.9 cP and gel strength ranged from 926.26 to 4945.99 dyne/cm².

1. Introduction
Seaweed is one of the commodities that acts as a major contributor to the production of the aquaculture sector. Every year Indonesian seaweed production continues to increase, from 2.574 million tons in 2009 to 3.082 million tons in 2010 so that Indonesia is also the largest producer of carbonated seaweed in the world, around 90% of the world market has been controlled by Indonesia [1]. Currently, the use of seaweed has progressed, which is made into agar-agar, alginate, carrageenan, and furselaran which are important raw materials for the food, pharmaceutical, cosmetics, and others. One type of seaweed that dominates Indonesia's exports is *Kappaphycus alvarezii*. [2]. *K. alvarezii* seaweed is leading commodity-producing carrageenan which is widely used in the paper, textile, photography, fish, and pasta canning industries. Indonesian carrageenan production reached 80%
(3,896 tons) and exported 3,156 tons in 2002. Whereas in 1996–2004, the value of Indonesia's carrageenan exports was relatively constant, with growth ranging from 2.49-2.92% per year [3].

Seeing the use and needs of carrageenan in various fields that are increasing, carrageenan production needs to be improved. There are various ways to produce carrageenan, one of which is the extraction process. In the last ten years, several alternative extraction techniques were introduced, including ultrasonic extraction [1]. The biggest advantage of carrageenan gel formation using ultrasonic extraction method is to maintain the quality of the gel texture, and the process is safer, simpler, effective and efficient. The use of waves with a frequency of 20-40 kHz can increase the texture properties of carrageenan gel, such as gel hardness. Whereas when using conventional extraction at the same yield it takes 3 hours and the amount of solvent and temperature is higher. In this study carrageenan extraction from K. alvarezii with the help of ultrasonic waves will be continued by optimizing contraction temperature and time and analyzing the effect of temperature and time on carrageenan quality on seaweed ratios with solvents 1: 30. quality carrageenan gel with characteristics and characteristics that are in accordance with the quality standards of carrageenan [3].

2. Methods
The method used is the extraction method using ultrasonic waves from two different sonication devices, namely, Elma sonicator. In the process of carrageenan extraction, the fixed variable for each extraction method used was pH 8.5-9, the frequency of ultrasonic waves 20-40 kHz and K. alvarezii seaweed were used as many as 15 grams in each experiment. [4].

Extraction with ultrasonic waves is carried out at a frequency of 40 kHz with variations in extraction: temperatures of 30, 40, 50, 60, and 70 °C; and time 20, 25, 30, 35 and 40 minutes to determine the optimum temperature which gives the maximum yield.

3. Result and discussion

3.1. % yield
% yield obtained with carrageenan quality based on FCC standards (Food Chemicals Codex), EEC (European Economic Community), FAO (Food Agriculture Organization) and Commercial analysis of water content, ash content, viscosity, and gel strength.

![Figure 1](image)

**Figure 1.** Relationship between time variation and extraction temperature with% of agency yield

In figure 1 it can be seen that the increase in temperature can cause an increase in yield, this is because the higher the extraction temperature, the termination of the polysaccharide chain bond becomes carrageenan. The extraction process with ultrasonic waves with a frequency of 40 kHz in this experiment showed that the highest carrageenan yield was achieved at temperatures and extraction times of 70°C for 30 minutes, namely 44.45% and the lowest carrageenan yield reached at temperatures and extraction times of 40°C for 20 minutes ie, 34%. Compared to the minimum standard of carrageenan yield determined by the trade department (1989) [5] (by 25%), the results still do not meet the standards.
3.2. Water content
Determination of water content contained in carrageenan powder products from K. alvarezii seaweed produced in this activity was carried out gravimetric according to the procedure issued by AOAC, (1984).

![Figure 2. Relationship between variations in temperature and time extraction to water content](image)

The carrageenan water content of K. alvarezii seaweed in this activity tended to increase with increasing time and extraction temperature, the highest water content was found in the temperature and extraction time of 70 °C for 40 minutes which was 14.18% and the lowest was at the extraction temperature and time 30 °C for 20 minutes which is 6.27%. The value of water content obtained meets the standard of carrageenan quality for commercial but based on carrageenan quality according to the FCC, EEC and FAO has exceeded the maximum water content of 12%. Increasing the moisture content of the temperature and extraction time can be caused by the extraction time giving enough time for the solvent to penetrate the cell wall and pull out the compounds contained in the material, resulting in yields with high water content.

3.3. Ash content
The value of ash obtained by carrageenan quality standards according to the FCC (Max. 35%) but based on carrageenan quality according to EEC (15-40%) and FAO (15-40%) are not included in the standard.

![Figure 3. Relationship between temperature and time extraction variations to ash content](image)

The carrageenan ash content of K. alvarezii seaweed in this activity tends to be stable even though with increasing time and extraction temperature, the highest ash content is found at temperature and
extraction time of 70 °C for 25 minutes which is 13.69% and the lowest is at temperature and time 60 °C for 40 minutes which is 12.52%.

3.4. Viscosity
Based on the results of the analysis of increasing time and extraction temperature does not affect the levels of carrageenan ash produced because the value is close to each other. Ash content is produced by burning organic matter and is closely related to the number of minerals in a material. The ash content is around 12.52-13.69%, this indicates that the amount of minerals contained in seaweed is relatively low.

The content of ash indicates the amount of mineral content in carrageenan which is not burned during the process of ignition. Seaweed includes food that contains high enough minerals such as Na, Ca, K, Cl, Mg, Fe, S, and trace elements, especially iodine [6].

Viscosity testing was carried out to determine the level of carrageenan viscosity on extraction time and temperature. Determination of the viscosity contained in carrageenan powder from K. alvarezii seaweed produced in this activity was carried out according to the procedure issued by AOAC, 1984, FMC Corp., 1977 using a Viscometer Brookfield.

![Figure 4. Relationship between temperature and time extraction variations to viscosity](image)

The carrageenan viscosity value of E. cottonii seaweed in this activity tended to decrease with increasing extraction time and temperature, the highest viscosity was found in the temperature and extraction time of 70 °C for 25 minutes which was 27.9 cP and the lowest was at temperature and extraction time 35 minutes is 10.4 cP. The viscosity values obtained meet carrageenan quality standards according to the FCC, FAO and commercial values, namely a minimum of 5 cP.

Increasing the time and extraction temperature affects the viscosity. The use of higher extraction temperatures causes more sulfate content to emerge from seaweed, causing viscosity to decrease. The increase in extraction time causes viscosity to decrease, this is due to the nature of the carrageenan viscosity is directly proportional to the sulfate content, where the long extraction time can reduce the levels of carrageenan sulfate so that the viscosity value also decreases.

3.5. Gel strength
Determination of gel strength contained in carrageenan powder from K. alvarezii seaweed produced in this activity was carried out according to procedures issued by AOAC, 1984 and FMC Corp., 1977.
Figure 5. Relationship between temperature and time extraction variations on gel strength.

The value of K. alvarezi seaweed gel strength in this activity tends to increase with increasing time and extraction temperature, the highest value of gel strength is found at temperature and extraction time of 70 °C for 35 minutes which is 4945.99 dyne/cm² and the lowest is at temperature and time extraction of 30 °C for 25 minutes is 926.26 dyne/cm², the value of gel strength obtained exceeds the carrageenan quality standard of commercial value, namely 685 ± 13.43 [1].

Based on the analysis of gel strength, the values obtained by the unstable increase level at each time variation and extraction temperature are more likely to increase with increasing time and extraction temperature so that it can be said that the higher the extraction time and temperature, the higher the gel strength.

Increasing the value of gel strength is related to the extraction time where the longer the extraction time, the lower the sulfate content, the higher the strength of the gel and this indicates a significant correlation between sulfate content and gel strength.

Conclusion
The results of extraction of K. alvarezi seaweeds are seaweed with a time variation of 40 minutes and the variable temperature is 70 °C, with the optimum value of temperature and extraction time is 70 °C for 30 minutes which produces carrageenan as much as 44.46%. The carrageenan quality test produced water content ranging from 6.27-14.18%, ash content 12.33-13.69%, viscosity of 10.4-27.9 cP and gel strength ranged from 926.26 to 4945.99 dyne/cm².

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