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## Extraction refined carrageenan using ultrasonic irradiation in from *Kappaphycus Alvarezii* originated from Lontar

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# Extraction refined carrageenan using ultrasonic irradiation in from *Kappaphycus Alvarezii* originated from Lontar

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**Abstract.** *Kappaphycus alvarezii* or commonly known *Euचेuma cottonii* is a source of kappa-carrageenan and could find cultivated in the coastal areas of Indonesia especially in Lontar Banten province. *Kappaphycus alvarezii* is one type of red seaweed contained kappa carrageenan. Lontar beach in Banten province is one that produced red seaweed In Indonesia. Refined Carrageenan could be used as a stabilizer, thickener, gel, medicine, cosmetics, etc. An important factor affected the production of refined carrageenan one of them is the temperature and extraction method. In this study refined carrageenan extracted used the ultrasonic irradiation method from *Kappaphycus alvarezii* originated from coastal lontar has never been studied. This research aimed to determined the effect of temperature using ultrasonic irradiation. The result indicated that higher temperature increased of the sulphate content, gel strength, gelling point and ash content but reduced in moisture and yield. This study the best temperature extraction is 700°C.

## 1. Introduction

Carrageenan are economically important biopolymers used in the pharmaceutical, chemical, and food industries. Indonesia is the first produced seaweed *Euचेuma cottonii* in world. The Total production of *Euचेuma cottonii* Indonesia reached 11 million tonnes and increased to 13.4 million tonnes in 2017 [1]. *Euचेuma cottonii* is a type of red seaweed (Rhodophyceae), several types of red seaweed have been identified, there is three types (k-,  $\gamma$ -, and i-carrageenan) are commercially available, *Euचेuma cottonii* has a macro part there is protein 5.12%, fat 0.13%, carbohydrate 13.38%, fiber 1.39%, ash 14.21%, water 12.9%, and Carrageenan 65.7% [1]. Carrageenan is one of the ingredients that could be founded in seaweed *E. cottonii* [2]. The product of refined carrageenan that extracted from red seaweed could be used as a stabilizer, thickener, gel, medicine, cosmetics, processed food, textile, air freshener and emulsifier [3]. Based on the structure of carrageenan it could be divided into 3 factions, based on its constituent there is Kappa, Iota and Lambda carrageenan. Refined Carrageenan (RC) is a processed from seaweed *Euचेuma cottonii* whose structure is Kappa Carrageenan. Carrageenan is a hydrocolloid compound consisted of the ester of potassium sodium, magnesium and potassium sulphate [4]. Carrageenan is extracted from *E. Cottonii* thus forms polysaccharides 3-linked-b-D-galactopyranose (G units) and 4-linked-a-D-galactopyranose (D-units) that contained 22% to 35% of the sulphate group. In the process of seaweed, extraction is known that temperature is very influential against the percentage of the yield. The conventional extraction methods, which already used for decades, require prolonged extraction times, and relatively larger quantities of solvent. Ultrasound-assisted extraction technique that can be offered high reproducibility in a shorter time, higher yields of bioactive compounds, the



simplified extraction process, decreased the temperature, reduced solvent consumption, and lower energy [5].

## **2. Methodology**

### *2.1. Carrageenan extraction*

In this study there were divided into three-part, the processed begun with the seaweed pretreatment then followed extracted used KOH. Continued with separated the filtrate from the sediment. The filtrate was taken and purified using 96% ethanol to form hydrocolloid fibers. Then the hydrocolloid fiber is neutralized and dried until a sheet is formed. The refined carrageenan can be obtained by grinding or size reduction. The final process is analyzing the physical and chemical properties of refined carrageenan. The next step is Immersed the sample with distilled water for 15 minutes. The sample is extracted with KOH for one hour. The filtrate separated from seaweed pulp and collected the filtrate. The filtrate is mixed with ethanol solution and stir it until the hydrocolloid fibers are formed. Then neutralized the filtrate used distilled water and the hydrocolloid fiber that formed dried used an oven.

### *2.2. Viscosity measurement*

Viscosity was measured using a Brookfield viscometer by dissolved 1.5 grams of sample into 100 ml of distilled water then heat the solution and stir it to a temperature of 75°C. the viscosity of the solution is measured when the temperature of the solution is 75°C. The viscosity value is known by reading the viscometer on a scale of 1-100 [6].

### *2.3. Gel strength measurement*

Carrageenan Edible Film is made by dissolved 1.5 gram RC with distilled water then heated on the hot plate until the temperature reached 80°C for 15 minutes. Then poured into a petri dish of 0.2 mL / cm<sup>2</sup>. Edible films that have been formed are left at room temperature for 2 hours. Then dried at 50°C for 24 hours. Edible films that formed are tested using the Chun yen tool with strong specifications pulling a 10 mm distance and a 0.5 mm/sec test speed [6].

### *2.4. Moisture content measurement*

RC samples were weighed 1 gr dried in an oven at 105°C for 30 minutes. The sample was cooled in a desiccator for 10 minutes until a constant weight was obtained and expressed as a percentage by weight of the sample [7].

### *2.5. Ash content measurement*

1 gram of carrageenan was weighed then it was spilled in a furnace at 600°C for 1 hour then weighed [7].

### *2.6. Yield testing measurement*

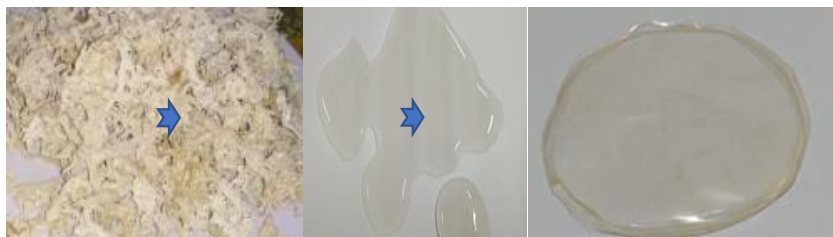
The yield is calculated based on the weight percentage of the carrageenan powder produced (A) on the weight of seaweed that used (B) [7].

### *2.7. Sulfate content measurement*

1 gram RC was added with 50 ml 0.2 N HCl and boiled for 15 minutes. 10 ml of 0.25 M BaCl<sub>2</sub> solution was added to the water bath for 5 minutes. The solution is cooled for 5 hours, the precipitate formed is filtered with filter paper and washed with boiling distilled water until it is free from chloride, then burned in the furnace at 700°C for 1 hour. The weight of white ash is the weight of BaSO<sub>4</sub> [7].

### 3. Results and Discussion

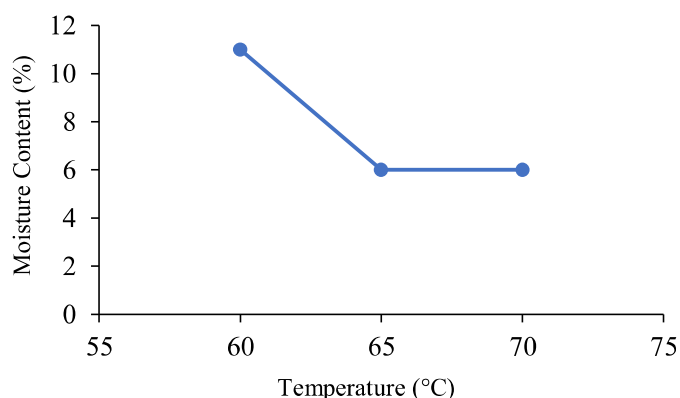
The experimental results from this study is presented in Table 1 which present chemical and physic analysis of RC product from seaweed *Eucheuma cottonii* that extracted using ultrasonic irradiation by temperature extraction variations.



**Figure 1.** Refined Carrageenan product.

#### 3.1. Moisture Content

Moisture content is intended to knowing the water content contained in the refined carrageenan product. The moisture content in the carrageenan is very important in the shelf life and durability of the material to be maintained so that the product could have a long shelf life.

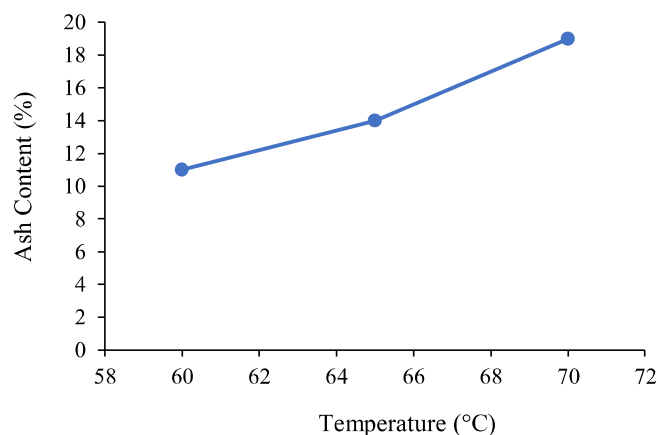


**Figure 2.** Moisture content vs temperature.

The moisture content of a refined carrageenan (RC) product is influenced by the temperature of the extraction that can be seen in Figure 2. The higher temperature extraction it would cause the mixture between the solution of KOH with seaweed will occur clotting in the results. This is due to the *Eucheuma cottonii* type of seaweed that belongs to the Kappa carrageenan will form a strong gel in the potassium salts solution and its getting more agglomerated when the temperature is increased. Clotting in RC caused surface area became smaller that caused there is still an amount of water that trapped in RC product in conventional Extraction [8]. But in ultrasonic irradiation, the clotting did not occur by increased the temperature of extraction because the extraction in ultrasonic irradiation is with cavitation bubble that directed to the molecule of *Eucheuma cottonii*.

#### 3.2. Effect of temperature on ash content

The ash content testing on RC aims to determine the mineral levels of the remaining inorganic substances. The average result of the analysis showed that different temperature treatments affected the ash content. This is because higher the Temperature could caused cation +K reacted with carrageenan that resulted higher in ash content The high level of ash in the RC product is also due to the mineral element that is not soluble during extraction that is calcium [9].

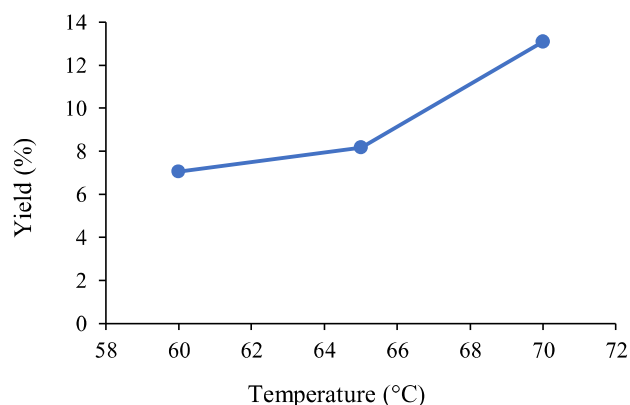


**Figure 3.** Ash content (%) vs temperature (°C)

### 3.3 Effect of temperature on yield of the carrageenan

The yield is compared to the weight of RC products that obtained against the weight of the seaweed that used in extraction. The RC yield calculation is based on the dry weight of the product which means that the yield represents the efficiency value of the processing process until it can be known the amount of carrageenan that produced from the raw material [9].

The increased temperature extraction caused higher cavitation bubble that formed that made the ultrasonic irradiation is more effective so the mass transfer of sulfate that removed is more and accelerate the formation of 3,6-Anhydro-galactose during the extraction process. Increase in temperature can also lead to increased solvent solubility and can enlarge the pores of solids so that the solvent of KOH could enter through the pore solids and dissolves the solids component that trapped in the form of sulfate.



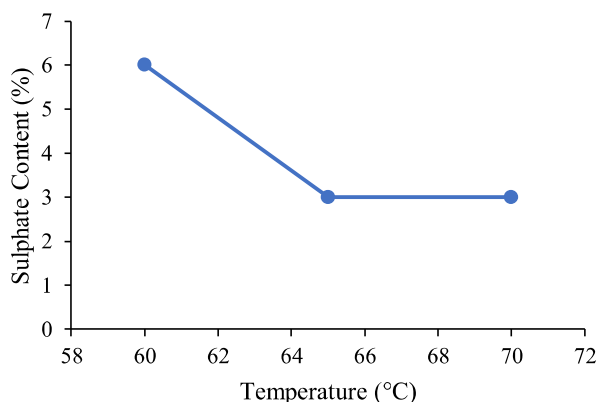
**Figure 4.** Yield (%) Vs Temperature (°C)

### 3.4 Effect of temperature on sulfate content

The sulfate content is expressed as the degree of substitution which is the mean number of sulfate per disaccharides repeat unit in carrageenan. Carrageenan can be classified in three ways, based on the amount and position of sulfate groups and its properties. Testing of sulfate rate aims to find out how much residual sulfate cannot be extracted by the KOH solvent. This research shows that the higher the

temperature then the sulfate rate is getting down. The decrease in the sulfate rate affected the physical properties of the product. This research is in line with [10] that the content of sulfate on the matter is very influential about the formation of 3.6 anhydrous-galactose, low sulfate will increase the content of 3.6 anhydrous galactose.

The increasing 3.6 of the galactose Anhydro will increase the value of the gel strength [7]. Sulfate rate is directly proportional to the viscosity due to the rejection of negative ester sulfate charges resulted in a chain of molecular chains. The molecular chains are a strain due to the hydrophilic properties of a polymer surrounded by a demobilized water molecule, caused a condensed solution to be viscous [10].

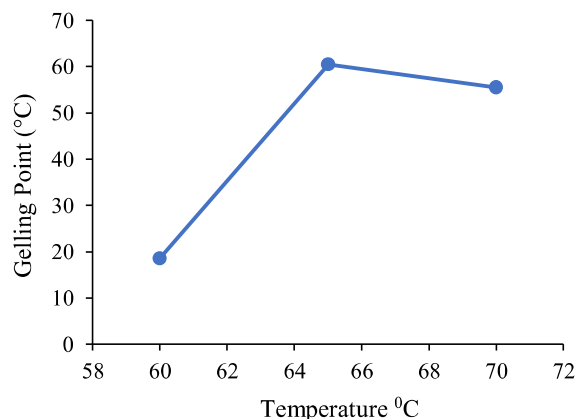


**Figure 5.** Sulfate Content (%) Vs Temperature ( °C)

### 3.5 Effect of temperature against a gelling point

The temperature of the gelling point is the temperature of the solution that is in a certain concentration begins to form the gel. Carrageenan can form a reversible gel, which means forming a gel at the time of cooling and melting back if heated [11].

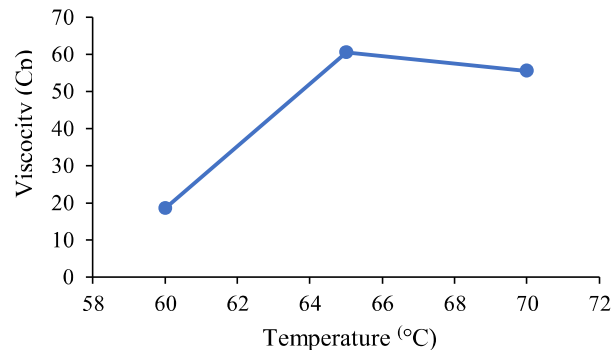
the higher temperature would facilitate the solution of KOH to remove the sulfate levels contained in the seaweed. This is in line with the Friedlander and Zelokovitch quoted [12] that the gelling point is directly proportional to the content 3.6-anhydrous-galactose and inversely proportional to the content of the Sulphate. The presence of sulfate tends to cause sol-shaped polymer so that the gelling point is difficult to form.



**Figure 6.** Gelling point (%) vs temperature (°C)

### 3.6 Effect of temperature on viscosity

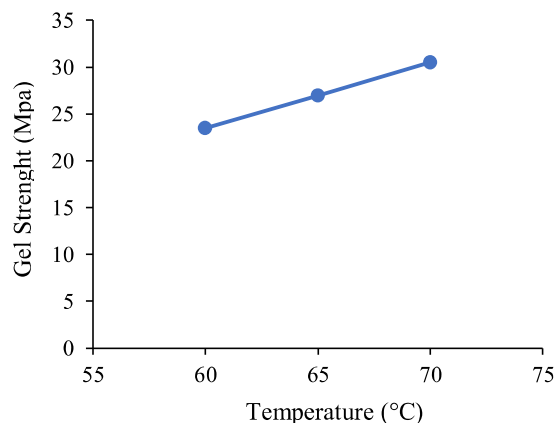
The quality of the carrageenan also depends on the Viscosity of the carrageenan. Viscosity increases exponentially with a higher molecular weight and/or higher solution concentration. Viscosity decreases with increased in temperature and vice versa. Decreased the sulfate content that caused establishment of a bond of 3,6 anhydrous –D galactose because of ion +K capable to eliminate sulfate bond. The decrease sulfate content it will decrease the viscosity because it causes the more bonds 3.6-anhydrous-D-Galaktosa are formed, it will increased the gel strength [13].



**Figure 7.** Viscosity (%) vs temperature (°C).

### 3.7 Effect of temperature on gel strength

The main of physical properties of refined carrageenan is gel strength because the gel strength demonstrated the ability of the carrageenan in gel formation. The strength of the gel is affected by the content of sulfate and 3.6-anhydrous-D-galactose. According to [7] The value of sulfated sulfate levels is directly proportional to the value of viscosity and inversely proportional to the strength of the gel. The smaller the sulfate rate the more the gel strength increases.



**Figure 8.** Gel strength (%) Vs temperature ( °C).

**Tabel 1.** The comparison the experimental results with the standard of product RC.

Parameters	Commercial standard	FAO	ECC	Best Result 70°C 1 Jam
Gel strength (gr/cm <sup>2</sup> )	685.5	-	-	30,5
Viscosity (cP)	5	-	-	55,45
Melting point (°C)	50	-	-	
Gel point (°C)	max.34	-	-	30
Ash content (%)	min 18.6	15-40	15-40	19
Water content (%)	Maks.12	Maks.12	Maks.12	6
Yield (%)	min. 25	min. 25	min. 25	13
Sulfate content (%)	max.30	15-40	15-40	3

The best result in this study is with temperature extraction 70 °C the result is close to the standart FAO and ECC it could be seen in tabel 1. except for yield and sulfata content that below the standart.

#### 4. Conclusion

Based on this study can be concluded that the temperature extraction 70°C produced the best quality of RC although there are parameters that still not standard that need to improve.

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