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The effect of Temperature Drying of Seaweed, Addition of Calcium Hypochlorite, and Potassium Hydroxide on the Quality of Semi-Refined Carrageenan Products

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ABSTRACT

Indonesia is a major producer of Eucheuma cottonii seaweed, yet its utilization remains suboptimal. Approximately 80% of seaweed exports consist of raw materials with low market value. The research aims to enhance the quality and market value of seaweed by transforming it into semi-refined carrageenan products. The method used for carrageenan production involves extraction using a KOH solution with different concentration levels using 5%, 7.5%, and 10% (w/v), along with drying temperatures ranging from 80°C to 90°C. Additionally, bleaching treatment is applied using calcium hypochlorite with concentration level using 1.5% and 2.5% (w/v). The analyses conducted include water content, ash content, yield, viscosity, gel strength, and degree of whiteness. The optimal results for semi-refined carrageenan, based on yield analysis, water and ash content, gel strength, viscosity, and degree of whiteness, were obtained from samples with a KOH concentration of 10%, calcium hypochlorite concentration of 1.5%, and drying temperature of 80°C.

Keywords : *Bleaching, Ekstraksi, Eucheuma cottonii, Semi refined carrageenan*

1. INTRODUCTION

Indonesia, as an archipelagic nation consisting of 13,667 islands with a coastline spanning over 81,000 km, possesses a significant potential for seaweed cultivation. There are 782 species of seaweed in Indonesia, with only 18 types from 5 genera being traded domestically. However, Eucheuma and Gracilaria genera are extensively cultivated (Anggadiredja, 2011). Seaweed stands out as a promising commodity for domestic development due to its abundant availability, capable of stimulating both the coastal economy and the national economy. Seaweed has long been a key aquatic resource and held the fifth position in 2019 among exported marine and fisheries commodities (Ministry of Marine Affairs and Fisheries, 2020). Indonesia itself is a major producer of Eucheuma cottonii seaweed, yet 80% of its exports still consist of raw materials, specifically dried seaweed (Hikmah, 2015).

As one of the world's largest maritime nations, Indonesia is driven to develop seaweed cultivation, processing, and marketing (Hasan, 2019). An example of seaweed development is its transformation into carrageenan products, which have a higher market value compared to the selling price of dried seaweed. Carrageenan is utilized to enhance the stability of food materials in the form of suspensions or emulsions, serving as a stabilizer, emulsifier, and food thickener (Supriyanti et al., 2015).

Based on the research conducted by Uju et al. (2019), examining the influence of Peracetic Acid (PAA) as a bleaching agent on the characteristics of semi-refined carrageenan (SRC) from Kappaphycus alvarezii, it was found that the extraction process using 10% KOH and the addition of 0.5% PAA resulted in a lightness value of 80.46±0.01, with a yield of 23.50%, viscosity of 99.33 cP, gel strength of 307.63 g/cm2, sulfate content of 13.29% (w/w), and ash content of 8.33% (w/w). However, it is noted from this research that the addition of the bleaching agent PAA led to a decrease in yield, viscosity, gel strength, sulfate content, and ash content.

The commercial production of semi-refined carrageenan involves the following process stages: Firstly, raw seaweed is cleaned from sand, salt, and unwanted materials. Secondly, the seaweed is cooked or boiled in a potassium hydroxide (KOH) solution. Subsequently, the seaweed is rinsed or washed with flowing water until the pH of the seaweed washing water becomes neutral. The neutralized seaweed is then dried using sunlight. After the seaweed is dry, it undergoes grinding using a grinder, resulting in semi-refined carrageenan (Dewi et al., 2012). Currently, the city of Cilegon does not produce SRC, necessitating a more intensive study to enhance quality and efficiency in the process.

Based on the analysis of these issues, a research study was conducted to enhance the quality and market value of seaweed by transforming it into semi-refined carrageenan (SRC) products. Eucheuma cottonii seaweed was processed into semi-refined carrageenan using an extraction method with variations in KOH concentration, along with bleaching treatment using calcium hypochlorite ($Ca(ClO)_2$). Through this investigation, the aim is to achieve the best quality SRC in accordance with commercial standards

2. METHODS

2.1 Raw material and Equipment

The materials used in this research include distilled water, potassium hydroxide (KOH), calcium hypochlorite, and red seaweed of the Eucheuma cottonii type sourced from Lontar, Tirtayasa, Serang Regency. The equipment utilized comprises sieves, stirring rods, porcelain dishes, Erlenmeyer flasks, a furnace, beaker glasses, measuring glasses, grinders, hotplate magnetic stirrers, ovens, pH meters, thermometers, scales, Brookfield viscometer, and a water bath.

2.2 Methods

Firstly, the seaweed was cleaned from impurities and sand using clean water. Subsequently, 100 grams of seaweed are weighed for the extraction process. The seaweed is then extracted with a KOH Alkali solution, varying concentrations at 5%, 7.5%, and 10% (w/v), with a seaweed-to-alkali solution ratio of 1:10 (w/v). The extraction is carried out at a temperature of 90°C for 40 minutes using a hotplate. Afterward, the seaweed is drained before undergoing the bleaching process.

Bleaching is conducted using calcium hypochlorite at concentrations of 1.5% and 2.5% at room temperature for 90 minutes. The bleached seaweed is then neutralized with distilled water, and the pH is checked using a pH meter. The seaweed is subsequently dried in sunlight for 2 days, followed by further drying in an oven at temperatures of 80 °C and 90°C for 60 minutes each. After the drying process, the dried seaweed is ground using a grinder, and the carrageenan formed is sifted through a 60-mesh sieve. The resulting carrageenan is then weighed and subjected to analysis

2.3 Analysis

2.3. 1 Yield Analysis (FMC Corp, 1977)

Carrageenan yield is calculated based on the ratio between the weight of the produced carrageenan and the dry seaweed used.

$$Yield\ (\%) = \frac{Weight\ of\ carrageenan}{weight\ sample} \times 100 \quad \text{.............(1)}$$

2.3. 2 Moisture Content Analysis.

Moisture content analysis can be conducted by weighing 2.0 grams of carrageenan using a pre-dried porcelain dish for 1 hour at 105°C to ensure the dish is thoroughly dry. The dish containing carrageenan is then placed in an oven at 105°C for four hours, or until a constant weight is achieved. If A is the initial sample weight and B is the sample weight after drying.

$$Moisture\ content\ (\%) = \frac{A-B}{A} \times 100\% \quad \text{...............(2)}$$

2.3. 3 Analysis of Ash Content (AOAC, 1995)

The procedure for testing the ash content of carrageenan is as follows: firstly, a porcelain crucible is dried in an oven for 1 hour at a temperature of 105 °C and then cooled for 30 minutes in a desiccator. The crucible is weighed to determine the initial weight (A). Next, 1 gram of carrageenan sample (B) is placed into the porcelain crucible and then introduced into an electric furnace with a temperature of 600°C for 6 hours. Afterward, it is cooled in a desiccator, and upon reaching room temperature, the crucible and the cooled sample are weighed to obtain the final weight (C).

$$Ash\ Content\ (\%) = \frac{(A+B)-C}{B} \times 100\% \quad \text{...................(3)}$$

2.3. 4 Viscosity Analysis (FMC Corp, 1977)

In a viscosity analysis, the goal is often to quantify how easily a fluid flows under applied stress. This analysis is crucial for understanding the behavior of liquids and ensuring that they meet specific requirements for various applications, such as manufacturing processes, quality control, or product development. Techniques for measuring viscosity include viscometers or rheometers, and the results are typically reported in units such as centipoise (cP) or pascal-seconds (Pa·s).

The viscosity analysis can be conducted by heating a 1.5% concentration solution of carrageenan above a hot plate, with regular stirring, until the temperature reaches 80°C. The Brookfield viscometer is activated, and the viscosity of the solution is measured when the temperature of the solution reaches 75°C. The viscosity value is determined by reading the viscometer on a scale ranging from 1 to 100. Readings are taken after 8 complete rotations for spindle no.2 at a speed of 60 revolutions per minute (rpm). The readings are duplicated five times for spindle no.2 when expressed in centipoises (cP). The Brookfield viscometer is showed in Figure 1.

Fig. 1. Brookfield viscometer

2.3. 5 Gel Strength Analysis (FMC Corp, 1977)

The gel strength analysis typically involves assessing the firmness and cohesiveness of the gel. Various methods and instruments may be used depending on the nature of the gel. For example, in the food industry, a texture analyzer or a penetrometer might be employed to measure the force required to penetrate or deform the gel. 1.5 grams of dry carrageenan powder sample are dissolved in 100 ml of distilled water and heated with simultaneous stirring using a Hotplate Magnetic Stirrer at a heating temperature of 90°C for 20–30 minutes. Subsequently, the solution is stabilized with a water bath at a temperature of 80–90°C for 15 minutes to eliminate any waves. The solution is then poured into a plastic container, left undisturbed overnight at room temperature until it forms a gel. Once the solution has turned into a gel, the sample is removed and weighed. Afterward, the sample is analyzed using a texture analyzer.

3. RESULTS AND DISCUSSION

3.1 Effect of concentration of KOH, the concentration of calcium hypochlorite, and the drying temperature on quality of carragenan

The variations conducted include the concentration of KOH, the concentration of calcium hypochlorite (chlorine), and the drying temperature. The obtained results are then analyzed to determine whether the quality of the produced Semi-Refined Carrageenan (SRC) meets the standards set by FAO (Food and Agriculture Organization) and FCC (Food Chemical Codex). A comparison of data between the quality standards according to FAO and FCC and the analysis results from the conducted research is presented in Table 1.

Table 1. Comparison of SRC Analysis Results with FAO and FCC Quality Standards.

Variation			Analysis Parameters					
Drying Temperature	Chlorine (%)	KOH (%)	Yield (%)	water content (%)	ash content (%)	viscosity (cPs)	Gel Strength (g/cm^2)	White Degrees
80°C	1.5	5.0	29.54	11.55	30.67	45.83	88.5	78.15
		7.5	27.71	11.10	34.33	42.50	135.5	77.86
		10.0	27.56	9.00	38.67	37.50	297.5	78.85
	2.5	5.0	30.23	11.25	31.33	42.50	23.0	78.46
		7.5	26.71	11.05	35.33	36.67	46.0	78.83
		10.0	25.81	8.90	39.00	32.48	66.5	78.92
90°C	1.5	5.0	27.77	10.85	33.33	53.33	71.0	77.69
		7.5	25.48	10.10	35.67	40.83	103.5	77.45
		10.0	25.07	8.45	40.67	39.15	225.5	78.87
	2.5	5.0	28.18	10.10	34.67	44.17	59.0	79.46
		7.5	26.47	9.95	36.33	40.83	138.0	78.88
		10.0	25.53	8.55	41.67	34.98	179.0	78.96
Standard FCC			Min 25	Maks 12	18 - 40	Min 5	> 500	-
Standard FAO			Min 25	Maks 12	15 - 40	Min 5	-	-

Table 1 shows that the analysis results obtained meet both FAO and FCC standards. The highest yield is observed in the variation with 5% KOH, 2.5% calcium hypochlorite (chlorine), and a drying temperature of 80°C, amounting to 30.23%. This meets the FCC and FAO standards of a minimum of 25%. The moisture content varies from 8-11%, which is within the permissible range according to FAO and FCC standards, where the maximum allowed moisture content is 12%.

In addition, for ash content, the obtained values fall within the acceptable range. According to FAO, the allowed range is 15-40%, while according to FCC, it is 18-40%. The results comply with the standards, except for the variation with 10% KOH and a drying temperature of

90℃, where the obtained results do not meet the standards. The viscosity of the obtained SRC varies from 30-50 cPs, meeting the FCC and FAO standards, which specify a minimum viscosity of 5 cPs.

3.2 Effect of KOH Concentration on SRC Yield

Yield is one of the parameters indicating the quality and excellence of the carrageenan processing from seaweed, and the results of this research are illustrated in Figure 2.

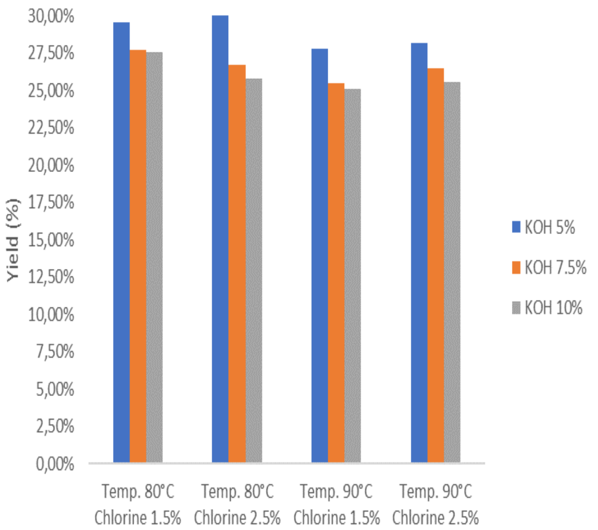


Fig. 2. Effect of KOH Concentration on Yield

In this study, a type of solvent, KOH, was used with concentration variations of 5%, 7.5%, and 10%. The selection of KOH as the solvent is due to its higher yield compared to NaOH. This is because the molecular weight of potassium in KOH is larger than the molecular weight of sodium in NaOH, resulting in a higher yield when using KOH (Distantina et al., 2012).

Figure 1 illustrates that at a 5% concentration, the yield is the highest compared to concentrations of 7.5% and 10%. This indicates that the higher the concentration of KOH used, the lower the yield obtained. This is attributed to the extraction process where carrageenan is released from seaweed due to the high concentration of KOH. This is evident in the extraction results, where seaweed extract transforms into gel when left undisturbed for some time. A study conducted by Arzani et al. (2020) also showed similar results, where the use of KOH concentrations of 6%, 8%, and 10% resulted in a decreasing yield with increasing concentrations of KOH used.

3.3 The Influence of KOH Concentration on the Moisture Content of SRC

The results of the moisture content analysis are presented in Figure 3.

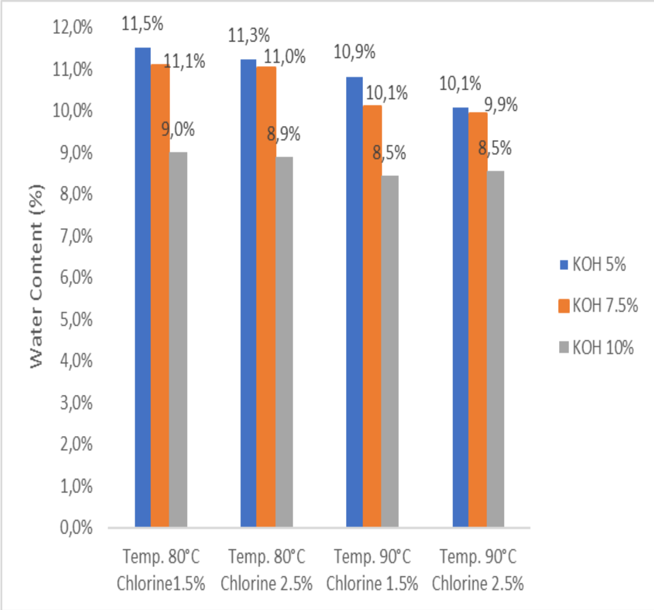


Fig. 3. The Influence of KOH Concentration on the Moisture Content of SRC

The moisture content is a crucial parameter determining the presence or absence of microbiological activity, which can influence the shelf life of carrageenan. The results of the moisture content analysis for SRC in this study reveal the highest moisture content in the 5% KOH concentration variation with drying temperature at 80°C and 1.5% calcium hypochlorite concentration, registering a moisture content of 11.5%. The lowest moisture content was obtained in the 10% KOH concentration with drying temperature at 90°C and 2.5% calcium hypochlorite concentration, yielding a value of 8.5%. This aligns with existing literature, where higher concentrations lead to lower moisture content. This is because the sulfate content, which binds water, decreases as it is reduced by the alkaline solution. Therefore, the greater the concentration of the alkaline solution, the more sulfate is reduced by the alkali (Hermanto, 2021).

Drying temperature is another factor influencing the moisture content. The higher the drying temperature, the lower the moisture content obtained. This is consistent with the results, where the moisture content at a drying temperature of 90°C is smaller compared to drying at 80°C. Higher drying temperatures affect moisture content because at higher air temperatures, a material's ability to release water from its surface increases (Fitriani, 2008).

3.4 The Influence of KOH on the Ash Content of SRC

The results of the ash content analysis can be shown in Figure 4.

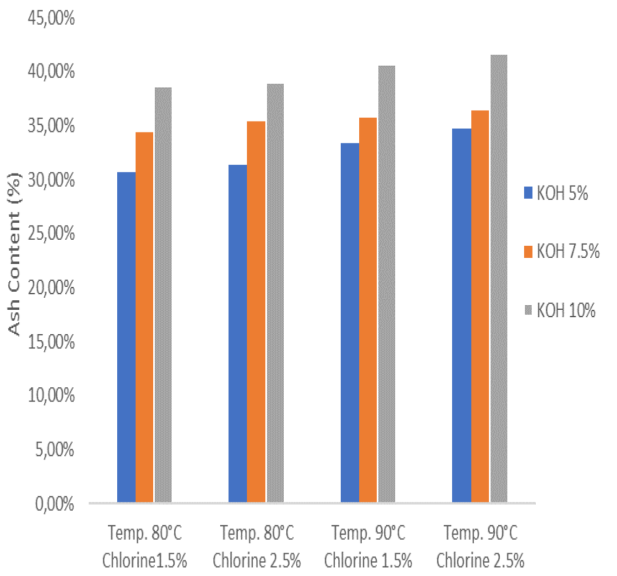


Fig. 4. The Influence of KOH on the Ash Content of SRC

Based on Figure 4, the ash content values increase with the increasing use of alkali concentration (KOH). Ega et al. (2016) state that the use of alkali solvents like KOH can increase ash content due to the increase in K+ ions interacting with carrageenan. This aligns with a study by Ega et al. (2016) using KOH solvent concentrations of 2%, 4%, 6%, 8%, 10%, and 12%, with the lowest result being 20.08% at a KOH concentration of 2% and the highest result being 33.68% at a KOH concentration of 12%. This statement is supported by Ningsih's research (2014), where an alkali solution of KOH with concentrations of 4%, 6%, and 8% and the addition of 1% KCl resulted in the lowest value of 38.88% at a KOH concentration of 4% and the highest value of 53.08% at a KOH concentration of 8%. This is in line with Basmal et al.'s statement (2005) that the increase in ash content is due to the presence of KOH solution, where K+ cations react with carrageenan.

3.5 Effect of KOH on the Viscosity of SRC

The results of the viscosity analysis can be seen in Figure 5. Based on Figure 5, the viscosity values decrease with the increasing concentration of alkali used (KOH). In a study by Tunggal et al. (2015), using KOH concentrations of 0.1 N, 0.3 N, 0.5 N, 0.7 N, and 0.9 N, the highest carrageenan viscosity was 275.3 MPa with 0.1 N KOH, and the lowest viscosity was 183.3 MPa with 0.9 N KOH. Furthermore, Hermanto's research (2021) on the extraction of red seaweed (Eucheuma cottonii) using $Ca(OH)_2$ alkali solution with concentrations of 0% (control), 5%, 6%, 7%, and 8% resulted in the lowest viscosity of 9.5 cPs with 8% $Ca(OH)_2$ and the highest viscosity of 27.5 cPs with 5% $Ca(OH)_2$.

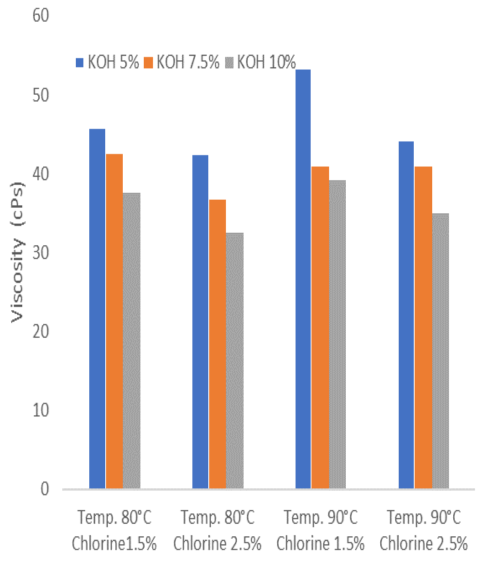


Fig. 5. Effect of KOH on the Viscosity of SRC

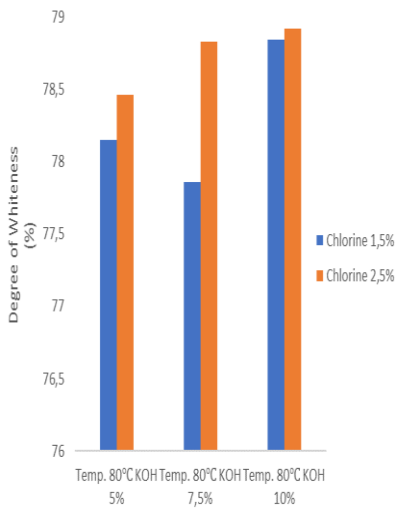
The decrease in carrageenan solution viscosity is due to the lower sulfate content, which, in turn, increases the gel strength. This is because the soluble salts in carrageenan reduce the net charge along the polymer chain. The reduction in charge leads to a decrease in repulsion forces between sulfate groups, weakening the hydrophilic properties of the polymer and causing a reduction in carrageenan solution viscosity (Basmal, 2005).

3.6 The Influence of Calcium Hypochlorite (Kaporit) Concentration on the Degree of Whiteness

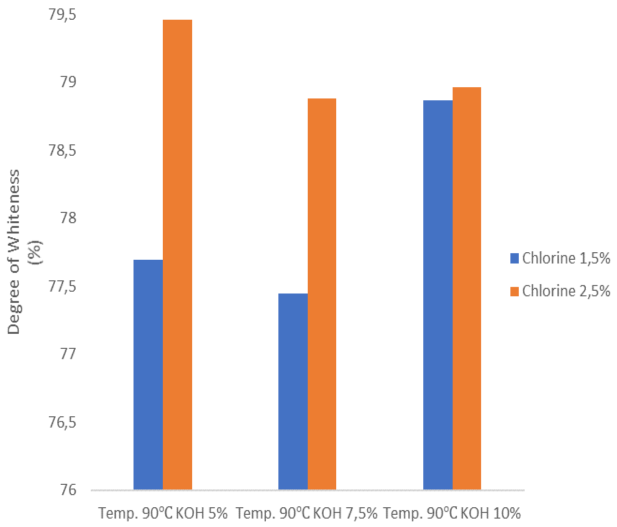
The results of the whiteness degree analysis are shown in Figure 6.

The degree of whiteness (L) indicates the brightness value of a material. The higher the L value, the brighter the measured material, approaching 100, while darker materials have L values approaching 0. Based on Figure 6 (a) and (b), the whiteness degree increases with the increasing concentration of the bleaching agent (Calcium Hypochlorite).

In a study by Uju et al. (2019) using Kappaphycus alvarezii seaweed treated with bleaching using peracetic acid and calcium hypochlorite at different concentrations (0.5%, 1.5%, and 2.5% w/w) for peracetic acid and 1.5% w/w for calcium hypochlorite, the whiteness degree using peracetic acid ranged from 80% to 86%, while with calcium hypochlorite, it was 74%. Therefore, increasing the concentration of peracetic acid resulted in higher whiteness degree values. In this study, the whiteness degree values obtained using calcium hypochlorite at concentrations of 1.5% and 2.5% (w/v) for red seaweed (Eucheuma cottonii) were approximately 77–78% and 78–79%, respectively.



(a)

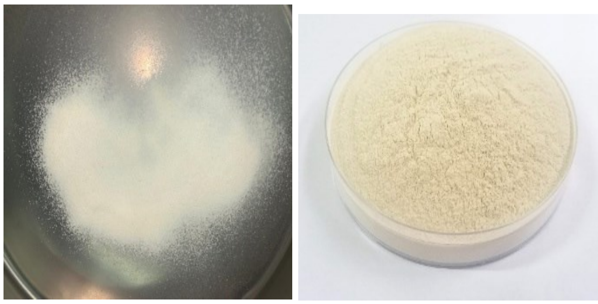


(b)

Fig. 6. The Influence of Calcium Hypochlorite (Kaporit) Concentration on the Degree of Whiteness (a). Temperature ekstraksi 80°C (b) Temperature ekstraksi 90°C

Red seaweed (Eucheuma cottonii) is primarily dominated by the red pigment phycoerythrin, even though, in nature, red seaweed can display other colors such as purple, dark brown, and dark green. According to Uju et al. (2019), the yellowish-brown color of semi-refined carrageenan is due to the presence of cellulose still present in SRC.

Based on Figure 7, it can be observed that the semi-refined carrageenan produced is off-white, while the commercial semi-refined carrageenan has a more creamy color. This indicates that the bleaching process using calcium hypochlorite produces a whiter color compared to commercial semi-refined carrageenan.



(a) (b)

Fig. 7. *Semi Refined Carrageenan* (a) This research (b) Comercial

3.7 The Influence of KOH on the Gel Strength of SRC

The results of the gel strength analysis of semi-refined carrageenan are presented in Figure 8.

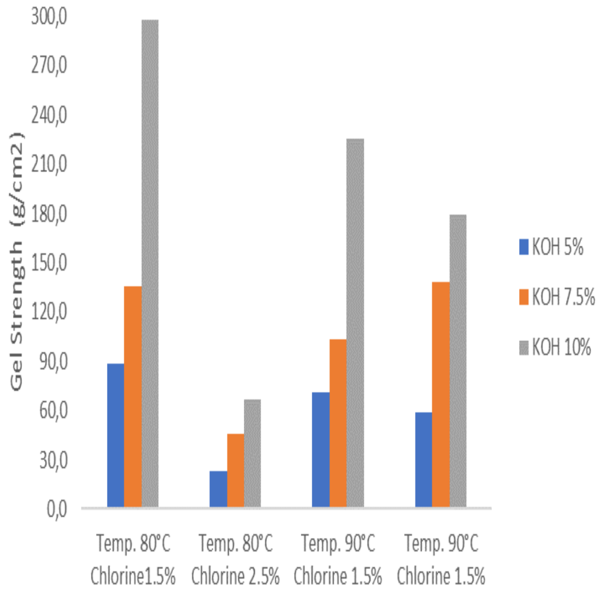


Fig. 8. The Influence of KOH on the Gel Strength of SRC

Figure 8 shows the fluctuations in the gel strength values obtained. In the variation with a temperature of 80°C and 1.5% calcium hypochlorite concentration, the highest gel strength value was obtained at a 10% KOH concentration, measuring 297.5 g/cm^2. In the variation with a temperature of 80°C and 2.5% calcium hypochlorite concentration, the highest gel strength value was obtained at a 10% KOH concentration, measuring 66.5 g/cm^2. Furthermore, in the variation with a temperature of 90°C and 1.5% calcium hypochlorite concentration, the highest value was obtained at a 10% KOH concentration, measuring 225.5 g/cm^2. Lastly, in the variation with a temperature of 90°C and 2.5% calcium hypochlorite concentration, the highest gel strength value was obtained at a 10% KOH concentration, measuring 179 g/cm^2. The gel strength results obtained align with existing literature, where higher alkali concentration values lead to larger gel strength values for carrageenan (Distantina et al., 2011). In a study conducted by Tunggal et al. (2015), increasing KOH concentrations ranging from 0.1 N to 0.9 N with an extraction time of 1 hour resulted in larger gel strength values with the increasing KOH concentration. According to FCC standards, the gel strength should exceed 500 g/cm^2. However, based on the results obtained for all variations, the gel strength does not meet the existing standards. This could be attributed to the rapid extraction process, resulting in gel strength analysis results that do not meet the standards.

4. CONCLUSION

The research results yield semi-refined carrageenan that complies with existing standards, namely the Food Chemical Codex (FCC) and the Food Agriculture Organization (FAO), based on yield analysis, moisture content, ash content, viscosity, and degree of whiteness. The most optimal semi-refined carrageenan is obtained in the variation with a 10% KOH concentration, bleaching with 1.5% calcium hypochlorite concentration, and drying temperature of 80°C. This is because the resulting gel strength value is the highest, and the use of different concentrations of calcium hypochlorite and drying temperature does not significantly impact the obtained results.

5. ACKNOWLEDGMENTS

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