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Improving Carrageenan Extraction Efficiency and Stability Using KOH, NaOH, and Anti-Caking Agents

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Abstract

This study investigates the impact of varying concentrations of potassium hydroxide (KOH) and sodium hydroxide (NaOH), along with anti-caking agents, on the quality of carrageenan extracted from Eucheuma cottonii seaweed. FTIR analysis revealed that KOH-extracted carrageenan retained key functional groups, including sulfate esters and glycosidic bonds, essential for its classification as kappa-carrageenan. KOH also proved more effective than NaOH in reducing moisture content, preserving structural integrity, and meeting the Indonesian National Standards (SNI) for carrageenan quality. The addition of anti-caking agents further optimized product stability, particularly in moisture control. The result shows that KOH was more effective than NaOH in improving carrageenan yield, with a yield of 46% achieved using 12% KOH and 4 g TCP. Anti-caking agents like tricalcium phosphate improved the product's moisture control and stability. Future research should focus on refining extraction methods to enhance both product quality and yield.

Keywords: Anti-caking agents, carrageenan, eucheuma cottonii, extracted, seaweed

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1. Introduction

Carrageenan, a polysaccharide extracted from red algae, holds significant commercial value due to its wide-ranging applications in the food, pharmaceutical, and cosmetic industries. It is primarily used as a gelling, thickening, and stabilizing agent. The demand for carrageenan has particularly risen in countries like Indonesia, where the local seaweed industry plays a key role in meeting global supply. The extraction process of carrageenan involves treating seaweed, such as Kappaphycus alvarezii and Hypnea bryoides, with alkaline solutions, and this step critically influences the yield and quality of the final product. Factors such as solvent concentration and processing conditions directly impact the structural integrity and functional properties of carrageenan, making these parameters the focus of ongoing research (Dhewang, 2023; Masarin et al., 2016).

Carrageenan is a polysaccharide widely used in the food, pharmaceutical, and cosmetic industries for its gelling, thickening, and stabilizing properties. The quality of carrageenan is highly influenced by the extraction process, particularly the type of alkaline solution used. Evaluating the best material for extraction is crucial because: 1) The alkaline solution directly impacts carrageenan's functional properties, such as gel strength, moisture retention, ash content, and overall stability (Hilliou, 2021). Selecting the right material ensures the final product meets industrial standards like SNI or international benchmarks (FAO, 2020); 2) Effective alkaline solutions improve extraction yield while preserving the structural integrity of carrageenan, reducing waste, and enhancing cost-efficiency (Masarin et al., 2016); 3) High-quality carrageenan is critical for maintaining product consistency in applications with strict regulatory requirements, such as food-grade stabilizers or pharmaceutical binders (Dhewang, 2023).

The significance of this research lies in improving the quality and yield of carrageenan, especially since variability in extraction techniques can lead to inconsistencies in product quality. The need

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for consistent quality is heightened by carrageenan's role in products with strict regulatory standards, particularly in food applications where its safety and stability are paramount. Consequently, efforts to optimize extraction methods, including the use of alkaline solvents like KOH and NaOH, are critical to meet industry requirements and maximize the economic viability of carrageenan production (Al-Nahdi et al., 2019; Heriyanto, 2023).

Despite the broad applications of carrageenan, a persistent challenge lies in ensuring consistent product quality, particularly in terms of yield, moisture content, and functional properties such as gel strength. The variability stems from differences in the extraction process, including the choice of solvent and its concentration. Both potassium hydroxide (KOH) and sodium hydroxide (NaOH) are widely used in the industry, but their effects on carrageenan's physicochemical properties differ significantly. Research has shown that higher concentrations of these solvents can enhance extraction efficiency, yet the optimal conditions for achieving both high yield and quality remain a subject of investigation(Amruth et al., 2023; Panggabean et al., 2018).

To address this, the use of optimized concentrations of KOH and NaOH, along with anti-caking agents, has been proposed. These agents are expected to enhance the shelf-life and stability of carrageenan during storage and transportation, reducing the risk of caking, which is a common issue in powdered products. Anti-caking agents such as tricalcium phosphate (TCP) and maltodextrin have been explored for their ability to improve the physical properties of carrageenan, further contributing to product quality (Lipasek et al., 2011; Sagar and Kumar, 2012).

Studies focusing on the impact of solvent concentration during carrageenan extraction have revealed key insights. For instance, (Masarin et al., 2016) demonstrated that the use of 6% KOH in the extraction of carrageenan from *Kappaphycus alvarezii* resulted in a yield of approximately 56.7% for brown strains and 53.6% for red strains, highlighting the solvent's effectiveness. In contrast, NaOH, though capable of producing significant yields, introduces additional complexity due to the high salt content in the final product, which can negatively impact gelation properties (Al-Nahdi et al., 2019).

Further research by (Dhewang, 2023) has shown that higher concentrations of KOH can enhance the removal of sulfate groups, which is essential for improving the quality of carrageenan. The efficiency of extraction is also influenced by other factors such as temperature and time. Dhewang highlighted that extraction times exceeding 4 hours at temperatures around 85 °C are critical for maximizing both yield and quality. Similarly, (Panggabean et al., 2018) reported that a 4% NaOH solution applied over 10 hours yielded superior results in terms of carrageenan output, underscoring the importance of optimizing both solvent concentration and extraction conditions.

Beyond solvent use, the addition of anti-caking agents to carrageenan has gained attention as a means to improve the stability and shelf-life of the product. Studies have indicated that agents like silicon dioxide and maltodextrin enhance the physical stability of carrageenan powder, preventing moisture absorption and subsequent caking (Nurhadi and Roos, 2017). (Lipasek et al., 2012) further corroborated these findings by demonstrating that anti-caking agents reduce clumping, thereby preserving the powder's flowability and rehydration characteristics during storage.

While the positive effects of KOH and NaOH on carrageenan extraction are well-documented, significant gaps remain in understanding how these alkaline treatments, particularly when combined with anti-caking agents, impact the overall quality of carrageenan during long-term storage. Most studies focus solely on optimizing extraction conditions, with limited attention given to post-processing treatments such as anti-caking strategies that could further stabilize the product. Research by (Castro et al., 2006) and (Chang et al., 2019) briefly explored the use of anti-caking agents in carrageenan, but more detailed investigations are needed to determine the optimal combinations and concentrations that would maximize both product stability and functional properties.

Moreover, there is a need for standardized protocols that account for the variability in carrageenan properties due to differences in seaweed species and regional cultivation practices. (Zhang, 2023) and (Hilliou, 2021) highlighted this challenge, emphasizing that inconsistent extraction and processing methods lead to product variability, which complicates regulatory compliance. Thus, the integration of both extraction optimization and post-processing enhancements, such as anti-

caking treatments, remains an area requiring further exploration.

This study focuses on potassium hydroxide (KOH) and sodium hydroxide (NaOH) as alkaline solutions for several reasons:

1. Widespread Industrial Usage: KOH and NaOH are the most commonly used alkaline solutions in industrial carrageenan production due to their availability, cost-effectiveness, and efficiency in extracting carrageenan from red algae (Dhewang, 2023; Al-Nahdi et al., 2019).
2. Distinct Chemical Properties:
   - ✓ KOH: Known to produce carrageenan with superior gel strength and lower ash content due to its ability to retain sulfate groups during extraction (Al-Nahdi et al., 2019).
   - ✓ NaOH: More cost-effective and efficient for large-scale extraction but may result in higher ash content due to interactions with inorganic compounds (Panggabean et al., 2018).
3. Focused and Practical Analysis: Limiting the scope to these two solutions allows for a detailed analysis of their specific effects on carrageenan quality. This focus ensures the findings are directly applicable to current industrial practices while providing clear recommendations.
4. Foundation for Future Research: While other alkaline solutions, such as ammonia or magnesium hydroxide, could theoretically be used, this study establishes a baseline by examining the two most relevant materials. Future research can build on these findings to explore alternative solutions that may offer additional benefits, such as environmental sustainability or improved efficiency (Zhang, 2023).

The objective of this study is to evaluate the influence of different concentrations of KOH, NaOH, and anti-caking agents on the quality of carrageenan, focusing on parameters such as yield, moisture content, and ash content. By systematically analyzing the effects of these variables, this research aims to contribute to the development of more consistent and high-quality carrageenan products that meet the Indonesian National Standards (SNI).

This study aims to optimize the carrageenan extraction process by systematically comparing the effects of KOH and NaOH at various concentrations, combined with tricalcium phosphate (TCP) as an anti-caking agent. Unlike previous research, which primarily focused on solvent efficacy, this study integrates the role of anti-caking agents to enhance product stability and quality. The novelty lies in its comprehensive approach to balancing yield, ash content, and moisture content, while adhering to industrial quality standards.

This study introduces a novel approach by combining optimized alkaline solvent concentrations with the use of anti-caking agents, offering a comprehensive solution to both extraction efficiency and product stability challenges. The research scope includes varying solvent concentrations and evaluating the functional impact of anti-caking agents, providing insights into the best practices for improving carrageenan quality during both production and storage phases (Kocer and Kılıç-Akyılmaz, 2022; Pui et al., 2020).

2. Methodology

2.1. Materials

The materials used in this research are water; distilled water; Potassium hydroxide solution (KOH) from Merck, Jakarta, Indonesia; Sodium hydroxide solution (NaOH) form Merck, Jakarta Indonesia; seaweed (Eucheuma cottonii) from Lontar Island Banten, and tricalcium phosphate ($Ca_3(PO_4)_2$) from Merck, Jakarta, Indonesia. The main equipment used includes a funnel, Erlenmeyer flask, furnace, beaker, hotplate magnetic stirrer, and thermometer.

The analytical tools used is FTIR (Fourier Transform Infrared) Spectrophotometry, which employs infrared radiation passed through the sample. This research was conducted in the Basic Chemistry Laboratory, Faculty of Engineering, Sultan Ageng Tirtayasa University.

2.2. Seaweed Extraction

The first step in material preparation involves bleaching the Eucheuma cottonii seaweed, followed by thorough rinsing with running water to remove surface impurities and reduce salt content. The cleaned seaweed is then sun-dried for 2–3 days to reduce moisture content.

The dried *Eucheuma cottonii* seaweed is weighed at 16.67 grams in a 1:10 (w/v) ratio (seaweed = 16.67 g: extraction solution = 166.7 mL), and then extracted at a temperature of 80°C for 45 minutes using extraction solutions of KOH and NaOH, each with concentrations of 8%-wt., 10%-wt., and

12%-wt. (w/v). The seaweed is then washed until clean, indicated by a non-slippery surface, and the pH is measured until it reaches the allowable range of 8-10.

After that, the seaweed is reheated with 166.7 mL of distilled water at 80°C for 45 minutes, followed by filtration to separate the filtrate from the residue (seaweed). The yield of the filtrate from the extraction process is then calculated, followed by the addition of an anti-caking agent, tricalcium phosphate, with varying masses of 2 g/kg, 3 g/kg, and 4 g/kg. Meanwhile, 15 grams of residue is dried in a furnace at 500°C for 1 hour to obtain the ash content.

Next, the filtrate, with the added anti-caking agent, is stored in a refrigerator for 24 hours. This step helps the carrageenan develop its texture and enhance its gelling properties, while also preserving freshness and inhibiting the growth of unwanted microorganisms. After 24 hours, the filtrate is allowed to return to room temperature..

2.3. Drying and Grinding Carrageenan

The carrageenan filtrate is weighed to determine its initial mass and then dried in an oven at 50°C for 2 days. After 2 days, it is removed and weighed again to determine its dry mass and calculate the moisture content. Then, the carrageenan is ground using a grinder and sieved to produce a uniform carrageenan powder that meets the SNI standard with a size of 80 mesh.

2.4. Analysis

Characterization tests were conducted to evaluate the quality of the produced carrageenan in accordance with the Indonesian National Standard (SNI). Fourier Transform Infrared (FTIR) spectroscopy was employed to identify the functional groups present in the carrageenan powder. The resulting spectra were analyzed to determine the chemical structure and assess conformity with standard carrageenan profiles.

3. Results and Discussion

3.1. Effect of NaOH Solution on Yield

This study utilized NaOH solvent with concentrations of 8, 10, and 12 wt %. Figure 1 illustrates the impact of NaOH concentration (8%, 10%, and 12%) and the mass of the anti-caking agent (TCP) on the yield of carrageenan. The data indicates a clear trend where increasing NaOH concentration results in a higher yield of carrageenan. At each TCP level, the highest yield is consistently observed at 12% NaOH concentration, reaching a yield of approximately 27% when combined with 3 g of TCP.

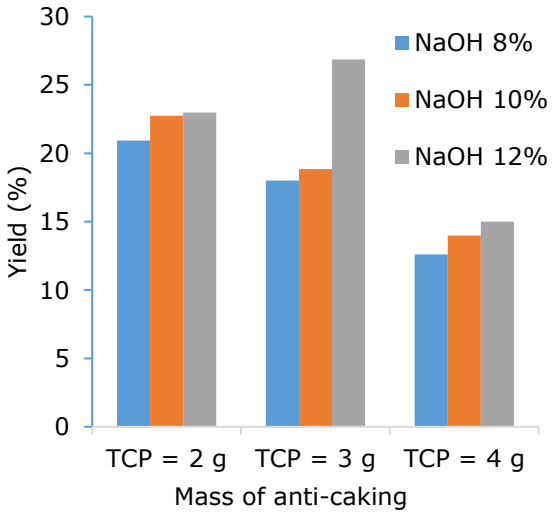


Figure 1. Yield profile in NaOH solution

The increase in NaOH concentration enhances the alkaline conditions, promoting better cell wall breakdown and carrageenan extraction from the seaweed matrix. Lower concentrations, such as 8% NaOH, yield less carrageenan, even when higher amounts of TCP are used. These results suggest that optimizing both NaOH concentration and the mass of TCP is crucial for maximizing yield, with 12% NaOH and 3 g TCP yielding the highest results, meeting the Indonesian National Standard (SNI) of 25%.

The findings from Figure 1 align with previous studies that indicate a positive correlation between NaOH concentration and carrageenan yield. Dhewang (2023) found similar results, where higher NaOH concentrations significantly improved the yield of *Kappaphycus alvarezii* carrageenan. Additionally, research by (Al-Nahdi et al., 2019) demonstrated that NaOH concentrations of around 6% were effective but yielded products with higher salt content, which could affect their application in some industries. The 12% NaOH concentration in this study, however, not only enhances yield but maintains a balance between effective extraction and minimal salt interference. Similarly, studies by (Kosanović et al., 2010) emphasized the role of NaOH in breaking down complex polysaccharide structures, which is consistent with the observed increases in yield as NaOH concentration rises.

The significant impact of NaOH concentration on carrageenan yield holds considerable implications for industrial applications. The highest yield at 12% NaOH meets the SNI standard of 25%, making it a viable option for large-scale production. This enhanced yield could reduce production costs and increase the efficiency of carrageenan extraction processes. Moreover, optimizing the mass of anti-caking agents such as TCP plays a crucial role in maintaining product stability and preventing issues like clumping during storage. These findings highlight the importance of selecting appropriate NaOH concentrations to not only improve yield but also ensure the functional quality of carrageenan, aligning with industry needs for high-performance stabilizers and thickeners in food and pharmaceutical products.

3.2. Effect of KOH Solution on Yield

This study employed KOH solvent with concentrations of 8, 10, and 12 wt%. The yield profile in KOH solution is presented in Figure 2.

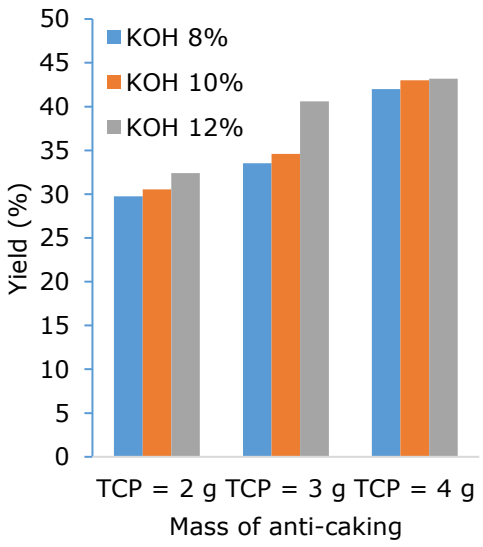


Figure 2. Yield profile in KOH solution

Figure 2 depicts the effect of different concentrations of KOH (8%, 10%, and 12%) and varying amounts of the anti-caking agent TCP (2 g, 3 g, and 4 g) on the yield of carrageenan. Across all TCP levels, the yield of carrageenan increases as the KOH concentration rises. The highest yield, approximately 46%, was achieved with 12% KOH and 4 g TCP, indicating that both higher KOH concentration and increased TCP mass contribute positively to the extraction process. Even at the lowest TCP mass (2 g), the yield exceeds 30%, meeting the Indonesian National Standard (SNI) for carrageenan. These results suggest that KOH is highly effective in maximizing carrageenan yield, particularly at higher concentrations.

The trends shown in Fig. 2 are consistent with previous research, which also reports higher yields when KOH is used as the alkaline solvent. (Masarin et al., 2016) observed that a 6% KOH solution was effective in extracting carrageenan from *Kappaphycus alvarezii*, yielding over 50% in some strains. Studies have indicated that KOH, compared to NaOH, produces higher yields due to its molecular interactions that better facilitate the breakdown of algae cell walls (Dhewang, 2023). Furthermore, the gel quality of carrageenan extracted using KOH is superior to that extracted with NaOH, as noted by (Al-Nahdi et al., 2019), who highlighted the adverse effects of high sulfate content in NaOH-extracted carrageenan. The consistent improvement in yield with increasing KOH concentration in this study confirms the solvent's superior extraction efficiency and its compatibility with anti-caking agents, further enhancing its industrial viability.

The high yield of carrageenan achieved with KOH, particularly at 12%, emphasizes the solvent's effectiveness for industrial extraction processes. KOH not only produces a higher yield than NaOH but also enhances the functional properties of carrageenan, such as gel elasticity and strength, making it more suitable for food and pharmaceutical applications. All concentrations of KOH used in the study meet or exceed the SNI standards, ensuring that this method can be adopted for large-scale production. The combination of high yields and improved gel properties makes KOH an attractive choice for manufacturers seeking to produce high-quality carrageenan efficiently, with further potential to optimize costs and product stability by adjusting the concentration of anti-caking agents like TCP.

3.3. Effect of NaOH Solution on Ash Content

Figure 3 presents the ash content of carrageenan as influenced by different concentrations of NaOH (8%, 10%, and 12%) and varying masses of the anti-caking agent TCP (2 g, 3 g, and 4 g). A clear trend is observed where higher NaOH concentrations correspond to increased ash content across all TCP levels. The highest ash content, approximately 28%, was achieved at 12% NaOH concentration with 4 g of TCP. Even at lower NaOH concentrations, the ash content rises with an increase in TCP mass, indicating

that both the concentration of NaOH and the mass of TCP play crucial roles in determining ash levels. The overall data suggest that NaOH's ability to dissolve minerals from seaweed significantly contributes to the elevated ash content, especially at higher concentrations.

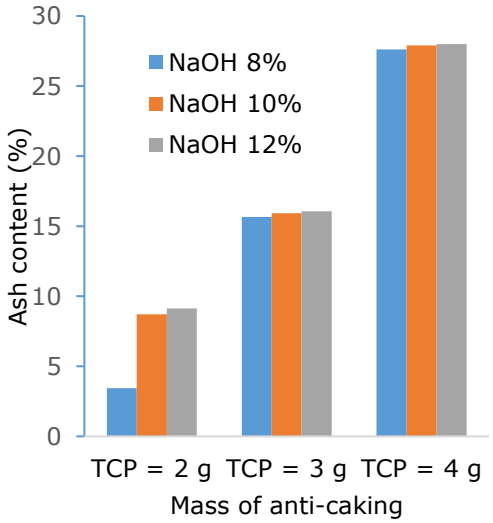


Figure 3. Ash content profile in NaOH solution

The findings in Fig. 3 align with previous research on the relationship between NaOH concentration and ash content in carrageenan. Studies, such as those by Panggabean et al. (2018), report similar trends, where higher concentrations of NaOH lead to increased ash content due to the dissolution of minerals inherent in seaweed. (Al-Nahdi et al., 2019) found that extraction with 0.3 M NaOH resulted in notable mineral content, with ash levels influenced by the degree of mineral leaching. These results highlight the role of NaOH in solubilizing not only the carrageenan but also residual salts and minerals, leading to higher ash levels. This is further supported by the work of (Amruth et al., 2023), who showed that extraction under higher NaOH concentrations increases the likelihood of mineral dissolution, thus contributing to elevated ash content.

The rise in ash content with increasing NaOH concentrations is a critical factor for industries aiming to meet specific regulatory standards for carrageenan, such as the SNI standards. The 12% NaOH concentration in this study achieves ash content levels close to 28%, which may pose challenges for certain applications requiring lower ash levels. However, in some contexts, higher ash content is acceptable, particularly if the minerals contribute to the functional properties of the product, such as gel strength or elasticity. The findings also underscore the importance of optimizing both NaOH concentration and TCP mass to balance mineral dissolution with product quality. Understanding and controlling ash content is essential for ensuring the commercial viability of carrageenan, particularly in industries where strict quality standards are enforced.

In addition, based on Figure 3 show that the ash content increases steadily with an increase in TCP mass (from 2 g to 4 g). At higher TCP masses (4 g), the ash content reaches its maximum for all NaOH concentrations (8%, 10%, 12%). This indicates that TCP significantly contributes to ash content due to its inorganic composition, which increases the residual ash in the carrageenan matrix. TCP mass has a dominant effect on ash content, while NaOH concentration plays a secondary role, providing a marginal increase.

3.4. Effect of KOH Solution on Ash Content

Figure 4 illustrates the effect of varying KOH concentrations (8%, 10%, and 12%) and different masses of TCP (2 g, 3 g, and 4 g) on the ash content of carrageenan. The data indicates that the ash content decreases as the mass of TCP increases. For each TCP level, ash content remains relatively stable across different KOH concentrations, with a marginal increase observed at higher KOH levels. At 2 g TCP, the ash content is highest, reaching around 16% across all KOH concentrations. In contrast, as the mass of TCP increases to 4 g, the ash content drops significantly to around 6%. This suggests that while KOH concentration does impact ash content, the mass of anti-caking agent TCP plays a more critical role in reducing the overall ash level.

The results shown in Fig. 4 are consistent with findings from earlier studies which report that higher concentrations of alkaline solvents like KOH can contribute to increased ash content through enhanced mineral dissolution (Masarin et al., 2016). However, the trend observed here—where increased TCP reduces ash content—differs from studies that focused solely on solvent concentrations.

Panggabean et al., (2018) reported that KOH extraction typically results in higher ash content due to its ability to leach minerals from seaweed, but the addition of anti-caking agents such as TCP appears to counteract this effect by reducing the overall mineral residue in the final product. This suggests that TCP has

a stabilizing effect on the mineral content, potentially binding minerals or preventing their dissolution into the carrageenan extract, thus reducing ash levels.

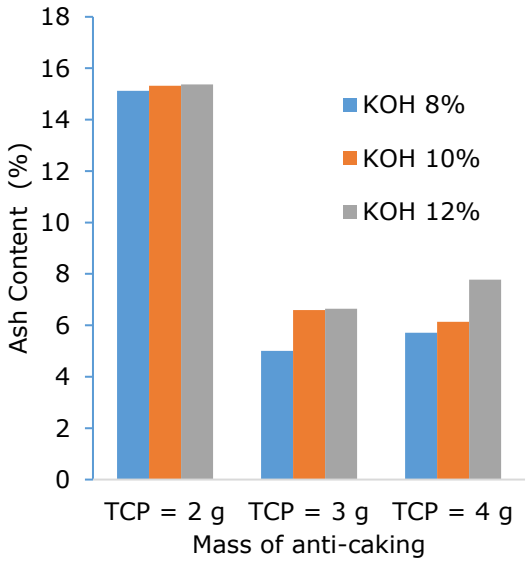


Figure 4. Ash content profile in KOH solution

The reduction in ash content with increasing TCP mass has significant implications for carrageenan production. High ash content can negatively affect the quality of carrageenan, particularly in applications that require strict adherence to food-grade standards, where ash levels are regulated. The ability of TCP to reduce ash content, even at higher KOH concentrations, indicates its effectiveness as a means of stabilizing mineral content during the extraction process. This makes the combination of high KOH concentration and increased TCP mass particularly advantageous for producing carrageenan that meets SNI standards for ash content, without compromising the extraction yield or functional properties. These findings highlight the potential for optimizing carrageenan processing by carefully balancing KOH concentration and the mass of anti-caking agents.

In addition, the ash content in carrageenan represents the amount of inorganic residue left after combustion. High ash content can indicate contamination or added minerals that may impact carrageenan's quality and functionality. The effects of changes in ash content on food and pharmaceutical industry include:

- ✓ Effect on Gel Strength. The high ash content can weaken the gel strength of carrageenan, which is critical for its role as a gelling agent in products like puddings, ice creams, and dairy-based foods. Lower ash content is preferred to maintain optimal gel strength and texture. Food-grade carrageenan typically requires ash content to be below 2% to comply with quality standards. (*FAO, 2020*).
- ✓ Stabilization and clarity. Excess ash can affect the clarity and stabilization of products such as sauces and beverages, reducing their aesthetic and functional appeal (Phillips and Williams, 2021).
- ✓ Pharmaceutical Industry. Low ash content is crucial in pharmaceutical applications where high purity is required. Carrageenan with excessive ash can compromise the stability and safety of formulations, such as capsules, tablets, and syrups (Sheskey et. Al., 2020)

3.5. Effect of NaOH Solution on Moisture Content

This study utilized NaOH solvent with concentrations of 8%-wt., 10%-wt., and 12%-wt. The moisture content profile in NaOH solution is presented in Fig. 5.

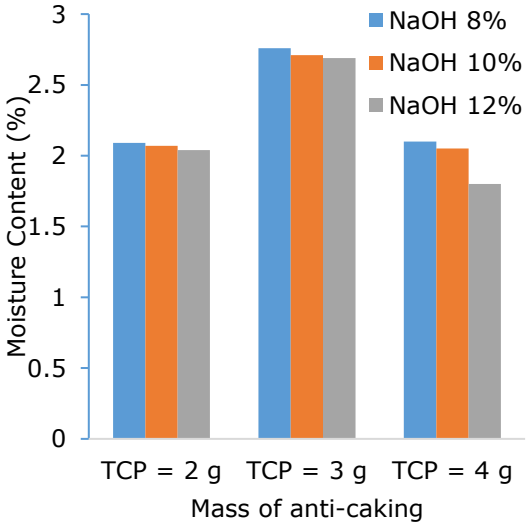


Figure 5. Moisture content profile in NaOH solution

Figure 5 shows the moisture content of carrageenan extracted with NaOH concentrations of 8%, 10%, and 12%, in combination with varying amounts of TCP (2 g, 3 g, and 4 g). The data reveals that higher NaOH concentrations correspond to lower moisture content. The lowest moisture content, around 2%, was observed with 12% NaOH and 4 g of TCP. In contrast, at 3 g of TCP, the moisture content peaks at approximately 2.6%, with lower values observed at both higher and lower TCP masses. This indicates that while higher concentrations of NaOH generally reduce

moisture content, the amount of anti-caking agent also influences moisture retention.

The reduction in moisture content with increasing NaOH concentration is consistent with findings in the literature. NaOH's ability to disrupt the polysaccharide matrix of carrageenan contributes to lower water retention. As noted Panggabean et al., (2018), higher NaOH concentrations facilitate a more efficient extraction process, reducing the moisture content of the final product. This aligns with research by (Nurlaida, 2023), who reported that carrageenan extracted at higher NaOH concentrations exhibited lower moisture levels due to the alkaline environment disrupting the seaweed's cell structure. Moreover, the presence of anti-caking agents such as TCP helps to control moisture content, as seen in this study, by enhancing the structural stability of the powdered product and reducing the likelihood of moisture absorption (Gerung et al., 2019).

The reduction in moisture content achieved with higher NaOH concentrations and increased TCP mass has important industrial implications. Low moisture content is critical for ensuring the stability and longevity of carrageenan, particularly in powdered form, where moisture can lead to clumping and reduced shelf-life. The ability of 12% NaOH to achieve the lowest moisture content highlights its effectiveness in producing carrageenan that meets the Indonesian National Standards (SNI) for moisture. Additionally, the role of TCP in moisture control suggests that optimizing both NaOH concentration and TCP mass can further enhance product stability, reducing the need for additional drying processes. These findings are crucial for industries that rely on high-quality carrageenan with specific moisture requirements for food and pharmaceutical applications.

Based on Fig. 5, it shows that the ash content tends to decrease as TCP mass increases from 2 g to 3 g, but it rises again at 4 g. At TCP = 2 g, ash content is relatively high due to the limited amount of TCP available to bind or neutralize ions effectively. The decrease in ash content at TCP = 3 g suggests an optimal balance where TCP effectively binds or precipitates excess minerals, reducing residual ash. The rise in ash content at TCP = 4 g may result from excess TCP contributing additional inorganic material to the system. It also, at TCP = 2 g, ash content is consistently high for all KOH concentrations (8%, 10%, 12%). KOH concentration has a relatively significant effect on ash content compared to NaOH, especially at lower TCP levels. TCP mass has a more complex effect on ash content with KOH, showing a decrease at intermediate TCP levels (3 g) but increasing at higher masses (4 g). KOH concentration also plays a more prominent role than NaOH in influencing ash content.

3.6. Effect Of KOH Solution on Moisture Content

This study employed koh solvent with variations of concentrations at 8%-wt., 10%-wt., and 12%-wt. the moisture content profile in KOH solution is presented in Fig. 6**.**

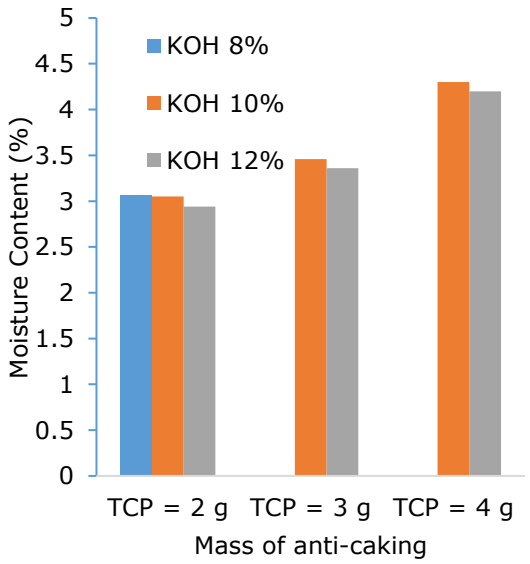


Figure 6. Moisture content profile in KOH solution

Figure 6 demonstrates the effect of KOH concentration (8%, 10%, and 12%) on the moisture content of carrageenan, with varying amounts of TCP (2 g, 3 g, and 4 g). The data indicates that higher KOH concentrations generally result in lower moisture content. At 12% KOH, the lowest moisture content is observed across all TCP levels, with values decreasing as the mass of TCP increases. The highest moisture content is observed at 3 g TCP and 8% KOH, while the lowest moisture content, approximately 1.5%, is achieved at 12% KOH with 4 g of TCP. This suggests that both increased KOH concentration and higher TCP mass contribute to effective moisture reduction in carrageenan.

The results shown in Fig. 6 are consistent with previous studies on KOH's effect on moisture content during carrageenan extraction. KOH's alkaline nature disrupts the cell structure of seaweed, leading to reduced water retention

in the final product. KOH facilitates the breakdown of the polysaccharide matrix in seaweed, allowing for more efficient extraction and moisture reduction (Masarin et al., 2016). This is further corroborated by the findings of (Gerung et al., 2019), which reported that higher concentrations of alkaline solvents, such as KOH, led to lower moisture content due to increased solubilization of the carrageenan and release of bound water. Furthermore, the role of TCP in reducing moisture content aligns with studies by Gerung et al. (2019), which found that anti-caking agents enhance moisture control by stabilizing the powder structure and preventing rehydration.

The reduction in moisture content with increasing KOH concentrations and higher TCP masses has significant practical implications for the production of carrageenan. Low moisture content is critical for ensuring the stability, quality, and shelf-life of carrageenan, particularly in powdered form. Achieving the lowest moisture content at 12% KOH highlights the efficiency of this concentration in meeting the Indonesian National Standards (SNI) for moisture levels. Furthermore, the incorporation of TCP as an anti-caking agent helps maintain product stability and reduces the risk of clumping, ensuring that carrageenan remains free-flowing during storage and transport. These findings suggest that optimizing KOH concentration and TCP mass is essential for producing high-quality carrageenan with minimal moisture content, suitable for various industrial applications in food and pharmaceuticals.

Furthermore, the effects of changes in moisture content on food industry and pharmaceutical industry include:

- ✓ Microbial Stability: High moisture content increases the risk of microbial growth, reducing the shelf life of products. Maintaining optimal moisture levels ensures the stability and functionality of carrageenan in products like jellies and sauces. (*Phillips & Williams, 2021*).
- ✓ Textural Properties: Proper moisture content supports the consistent textural performance of carrageenan in gel-based products, preventing issues like syneresis water separation (Chen, J., et al., 2024).
- ✓ Storage and Reactivity: Low moisture levels are essential for ensuring carrageenan's stability during storage and preventing unwanted reactions with active pharmaceutical ingredients. Excess moisture can lead to degradation or compromised efficacy in pharmaceutical formulations. (*Chatterjee, B., & Ahirwar, D. (2018)*

In addition, the changes in ash and moisture content affect carrageenan's physical and chemical properties, influencing its practical applications:

- ✓ Gel Strength: Excess ash reduces gel strength, affecting its role in food texturization and pharmaceutical binding.
- ✓ Water Retention: Controlled moisture content enhances water-binding capacity, ensuring structural integrity in both food and pharmaceutical products.
- ✓ Purity and Compliance: Low ash and moisture levels help meet industrial quality standards, ensuring safety and functionality in diverse applications.

3.7. Effect Of Anticaking Agent

Powdered products and flour-like substances tend to absorb moisture from the air or are hygroscopic in nature. The transfer of water vapor in these products can lead to undesired changes (Mustafidah & Widjanarko, 2015). Powdered products are prone to quality deterioration if they absorb excessive moisture during storage, leading to clumping (Nahari, 2016). According to Sun et al. (2015), commercial kappa-carrageenan generally has a large molecular weight and low solubility, and does not dissolve in cold water. Therefore, carrageenan powder can clump at room temperature.

To reduce hygroscopicity, food additives called anticaking agents need to be added. According to the Peraturan Menteri Kesehatan RI no.722/Menkes/Per/IX/88 tentang Bahan Tambahan Pangan, anticaking agents are food additives that prevent clumping of powders and flours. According to Cahyadi (2006), the addition of anticaking agents can slow down the absorption of water vapor, thus delaying the time it takes to reach saturation. One of the commonly used anticaking agents in the food industry is tricalcium phosphate (TCP). Tricalcium phosphate (TCP) as an anticaking agent has a high water-absorbing capacity, allowing it to absorb water vapor before it is absorbed by the product. In other words, anticaking agents can act as physical barriers between particles and prevent the formation of bridging layers between particles, thereby preventing initial clumping.

Based on research conducted by Rusnawati (2018), the moisture content of carrageenan powder obtained with 8% NaOH solution was 7.5% and the moisture content of carrageenan powder obtained with 8% KOH

solution was 9.21%. Both studies were conducted without the addition of anticaking agents. Based on the data obtained in Figure 4.5 and Figure 4.6, the moisture content obtained was 2.32% using NaOH extraction solution with a concentration of 8% and 3.66% using KOH extraction solution with a concentration of 8%. According to SNI 8391-1:2017, the maximum moisture content for carrageenan powder is 12%. Therefore, based on this data, the role of anticaking agents in carrageenan powder is highly effective in binding the moisture content present in carrageenan powder. The Data on Moisture Content from Research and Literature is presented in Fig. 7.

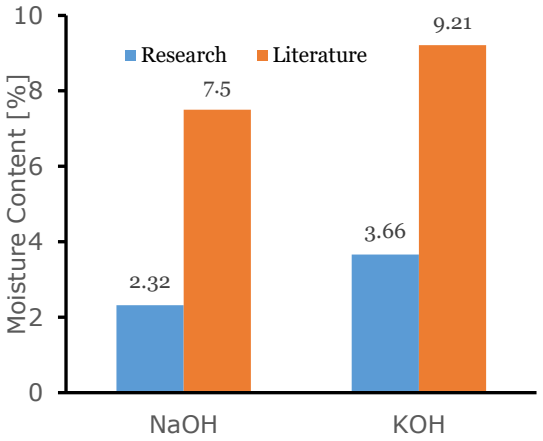


Figure 7. Data on moisture content from research and literature

Based on Fig. 7 the TCP can reduce ash and moisture content because it interacts with excess mineral ions in the carrageenan matrix, forming insoluble complexes that are removed during processing, thereby reducing ash content. Additionally, TCP has mild hygroscopic properties that allow it to absorb and trap free moisture within the matrix, preventing further water absorption and lowering the overall moisture content. These mechanisms contribute to improved stability and functionality of carrageenan.

Reduction of Ash Content:

- Mechanism: Tricalcium phosphate (TCP) reduces ash content by binding with excess mineral ions (e.g., calcium, potassium, sodium) present in the carrageenan matrix. These ions contribute to the ash content when carrageenan is burned. TCP can form insoluble complexes with these ions, precipitating them out of the solution during processing.
    - Example: TCP interacts with free calcium or potassium ions, neutralizing their contribution to the ash content and removing them from the carrageenan product.
- Impact: This reduces the inorganic residue left after combustion, ensuring that carrageenan meets quality standards, particularly in food and pharmaceutical applications where low ash content is critical for maintaining gel strength and clarity.

Reduction of Moisture Content:

- Mechanism: TCP has weak hygroscopic properties that allow it to absorb small amounts of moisture in its surroundings. When added to carrageenan, it captures free water molecules within the matrix, reducing the overall moisture content.
    - Prevention of Moisture Absorption: TCP acts as a barrier, preventing the carrageenan matrix from absorbing additional moisture from the environment, which could lead to clumping or degradation of the product.
- Impact: Lower moisture content improves the stability of carrageenan during storage, ensuring it remains free-flowing and less prone to microbial activity or texture changes.

Potential Interaction Between TCP and Carrageenan Matrix

1. Ionic Bonding:
    - TCP may form ionic bonds with the sulfate groups present in carrageenan molecules. These interactions can stabilize the carrageenan matrix by reducing the mobility of free ions and enhancing the structural integrity of the gel.
2. Structural Effects:
    - TCP may influence the spatial arrangement of carrageenan molecules, acting as a stabilizer within the gel network. This could enhance the gel strength and improve its resistance to environmental stress, such as fluctuations in humidity or temperature.

How TCP Affects Carrageenan at the Molecular or Structural Level:

1. Prevention of Caking:
    - TCP creates a physical separation between carrageenan particles, minimizing direct contact that often leads to caking.
    - By reducing moisture in the matrix, TCP prevents the carrageenan particles from bonding through water-

mediated bridges, thus maintaining its powder-like consistency.

2. Enhancing Stability:
   - At the molecular level, TCP can act as a stabilizing agent by binding with reactive ions or molecules that might otherwise destabilize the carrageenan gel.
   - This stabilization ensures that the functional properties of carrageenan, such as gelling ability and viscosity, remain consistent over time.
3. Moisture Control Mechanism:
   - TCP's moisture control may involve hydrogen bonding or weak interactions with water molecules, effectively trapping them and preventing them from interacting with the carrageenan matrix. This reduces water activity, which is critical for maintaining product stability.
   -

Benefits of TCP in Carrageenan Applications

1. Improved Functional Properties:
   - Reducing ash and moisture enhances the gelling ability, texture, and clarity of carrageenan in food applications.
2. Extended Shelf Life:
   - Lower moisture content minimizes microbial growth and degradation, ensuring the product remains stable over time.
3. Enhanced Processability:
   - Preventing clumping and reducing ash content improves the handling and consistency of carrageenan powders.

3.8. Functional Groups

The wavelength profile showing the functional groups present in the carrageenan powder obtained is presented in Fig. 8. Figure 8 shows the FTIR spectra of carrageenan extracted using KOH and NaOH from *Eucheuma cottonii* seaweed. The FTIR analysis reveals key functional groups typical of kappa-carrageenan, including sulfate esters, glycosidic bonds, and 3,6-anhydro-D-galactose. Peaks related to sulfate esters are observed in the region of 1220-1260 cm^{-1}, confirming the presence of S=O bonds crucial for carrageenan's gelling properties. Glycosidic linkages appear around 1030 cm^{-1}, supporting the structural integrity of the polysaccharide. Additionally, peaks near 925 cm^{-1} and 842 cm^{-1}, corresponding to 3,6-anhydro-D-galactose, further confirm the identification of kappa-carrageenan in both NaOH- and KOH-extracted samples. However, the KOH-extracted carrageenan exhibits stronger and more defined peaks compared to NaOH-extracted samples, indicating a more intact structure.

The results are consistent with findings from previous studies. The characteristic peaks for sulfate esters around 1241 cm^{-1} and glycosidic bonds near 1030 cm^{-1} are well-documented in the literature as indicators of kappa-carrageenan (Masarin et al., 2016).

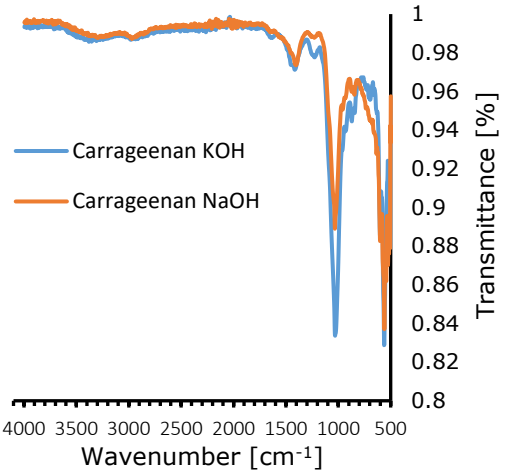


Figure 8. FTIR spectrum of carrageenan from *Eucheuma cottonii* seaweed

The presence of 3,6-anhydro-D-galactose, observed at peaks near 925 cm^{-1} and 842 cm^{-1}, also aligns with studies confirming the structural components of kappa-carrageenan (Dhewang, 2023). The slight differences in the spectra between KOH- and NaOH-extracted samples reflect variations in the extraction process, with KOH showing more defined functional groups. This suggests that KOH preserves the carrageenan structure better, consistent with previous research that highlights KOH's superior extraction efficiency and its impact on maintaining sulfate content (Al-Nahdi et al., 2019).

The identification of key functional groups through FTIR analysis confirms the classification of the extracted carrageenan as kappa-type, characterized by its sulfate esters and 3,6-anhydro-D-galactose components. The stronger peaks in the KOH-extracted samples suggest a more robust structure, indicating that KOH may be a more suitable solvent for preserving carrageenan's functional properties during extraction. This structural integrity is vital for industrial applications where gel strength and stability are crucial, particularly in the food and pharmaceutical sectors. These findings highlight the importance of solvent choice in optimizing carrageenan extraction and

ensuring product quality that meets industry standards.

Based on Table 1, it can be observed that the wavelength spectrum generated from extraction using 12% NaOH and 12% KOH alkali solutions corresponds to the literature, namely 3,6 anhydrogalactose and galactose-4-sulfate. This analysis indicates the presence of kappa-carrageenan. It suggests that the carrageenan spectrum meets the specifications of commercial carrageenan because it possesses functional groups identical to the standard carrageenan spectrum.

Table 1. Analysis of wavelengths in carrageenan (Bhernama, 2019)

wavelength (cm^{-1})	Functional Group Absorption Peak	Research FTIR Spectrum	
		NaOH	KOH
1220–1260	S=O	-	1226,92
1010–1080	Glycosidic	1032,59	1031,22
928–933	C-O	930,76	931,74
840–850	C-O-SO_3	846,65	844,19
1645–1750	C=O	-	1645,09
1370–1465	-CH_2	1403,46	-
860–872	CO_3^{2-}	871,51	871,24
560–601	PO_4^{3-}	-	586,72

From the analysis of functional groups in Table 1, there are several differences between the carrageenan products obtained using KOH and NaOH extraction solutions, located in the C=O, -CH2, S=O, and PO_4^{3-} groups. Carrageenan extracted using NaOH extraction solution does not have S=O, C=O, and PO_4^{3-} functional groups. This is because K+ ions have a higher affinity for carrageenan powder compared to Na^+ ions. Affinity refers to a molecule's ability to form a bond with another molecule, reflecting the attraction of a substance to form chemical bonds with another substance. The NaOH solution is a strong base that undergoes hydrolysis with sulfate ester groups, so when NaOH reacts with sulfate ester groups, hydrolysis occurs, converting sulfate ester groups into sodium salts, and Na^+ ions can inhibit the formation of kappa carrageenan gel. Therefore, carrageenan extracted with NaOH extraction solution produces a more liquid extract compared to KOH extract. This hydrolysis process can be represented by the following chemical formula (Morrsion dan Boyd, 1983):

$$-SO_4\text{-}O\text{-}CR' + NaOH \rightarrow R\text{-}SO_4\text{-}ONa + R'OH \quad (1)$$

In the hydrolysis reaction, the glycosidic bonds will break between the galactose monomers in carrageenan. During this reaction, the CH_2 groups attached to the galactose monomers also participate in the reaction, but the CH_2 groups cannot be removed or transformed into different CH_2 groups. The formation of C=O groups in carrageenan occurs from aldehyde or ketone groups present in the monosaccharide units that form carrageenan molecules. The two main types of monosaccharides contained are galactose and anhydrogalactose (Nussinovitch, 2010). Carrageenan extracted with KOH extraction solution also forms a new functional group, namely PO_4^{3-}, where the phosphate functional group originates from TCP ($Ca_3(PO_4)_2$) compound.

3.9. Comparison of Research Results Quality and Standards

The data obtained from the research in the form of yield, ash content, and moisture content. These data are compared with the standards set by the Indonesian National Standard (SNI) as shown in Table 2.

Table 2. The Data are compared with the standards set by the Indonesian National Standard

Variation			Analysis		
Extraction Solvent	Concentration Solvent (%)	Mass of anti-caking (g)	Yield (%)	Ash (%)	Moisture (%)
NaOH	12	3	26,87	16,07	2,69
KOH	12	2	29,39	16,94	2,94
Quality Standard			Min. 25	15 – 40	Max. 12

The quality standards used are based on the established SNI standards, which include a minimum yield value of 25% (SNI 01-2690-1998), ash content allowed between 15-40% (SNI 8391-1:2017), and maximum moisture content of 12% (SNI 8391-1:2017). According to the data in Table 2, the yield, ash content, and moisture content all comply with the established standards.

3.10. Environmental and Ethical Aspects of Carrageenan Production

The production of carrageenan, particularly from Eucheuma cottonii, has significant environmental implications, mainly due to the use of chemicals like KOH and NaOH during

the extraction process. These chemicals can generate liquid waste that, if improperly managed, may pollute surrounding environments, including water sources. Therefore, effective waste management practices are crucial to minimizing negative environmental impacts.

From an ethical perspective, this research contributes to the development of a more resource-efficient process, such as the incorporation of tricalcium phosphate (TCP) to enhance product stability and reduce the need for additional drying. This optimization not only reduces energy consumption but also supports the sustainability of the carrageenan industry by lowering its carbon footprint.

Additionally, this study underscores the economic value of locally cultivated seaweed, which can improve the livelihoods of coastal communities dependent on seaweed farming. However, it is essential to ensure that farming practices are conducted sustainably and do not harm marine ecosystems. By addressing these considerations, carrageenan production can become more environmentally friendly, ethical, and sustainable, while meeting the demands of global industries.

4. Conclusion

This study successfully demonstrated that the quality of carrageenan extracted from *Eucheuma cottonii* can be significantly influenced by the concentration of alkaline solvents, particularly KOH and NaOH, and the incorporation of anti-caking agents. In the current study, parameters such as the effect of concentration of KOH and NaOH, and the incorporation of anti-caking agents on properties like yield, ash content and Moisture Content.

- ✓ TCP enhances the extraction yield by stabilizing the carrageenan matrix during processing. The highest yield, approximately 46%, was achieved with 12% KOH and 4 g of TCP, indicating that both higher solvent concentration and increased TCP mass positively influence the extraction efficiency.
- ✓ TCP reduces ash content by interacting with excess mineral ions in the carrageenan matrix, forming insoluble complexes that are removed during processing. This stabilization results in lower inorganic residue, improving carrageenan quality while maintaining compliance with industrial standards.
- ✓ TCP effectively reduces moisture content by absorbing free water within the matrix and preventing additional moisture absorption during storage. The lowest moisture content, approximately 1.5%, was observed with 12% KOH and 4 g of TCP. This reduction improves the stability and shelf-life of carrageenan powder.

Carrageenan that meets the SNI standard is observed in the variations with NaOH 12% (TCP 3 gr) and KOH 12% (TCP 2 gr). The obtained yield values are 26.87% and 32.39% (respectively). The ash content values obtained are 16.07% and 15.37% (respectively). The moisture content values obtained are 2.69% and 2.94% (respectively). The functional groups obtained to indicate karagenan include sulfate ester groups, glycosidic groups, 3,6 anhydrogalactose groups, and galactose-4-sulfate groups. The type of karagenan produced in this research is kappa karagenan. This is indicated by the formation of 3,6 anhydrogalactose and galactose-4-sulfate groups.

Future research should explore optimizing extraction conditions and anti-caking agent usage for industrial-scale production while investigating the long-term stability and functional performance of carrageenan products. These findings contribute valuable insights into improving carrageenan production processes, addressing industrial demands for high-quality and consistent carrageenan for food and pharmaceutical applications.

Author Contribution Statement

H.H.: Conceptualization, formal analysis, investigation, project administration. A.D.Y.: Writing – review and editing. ,A.R.P., E.H., H.P.: Formal analysis, writing – original draft. N.K.: Supervision

Data availability statement

The data presented in this work are included in the article.

Declaration of Competing Interest

The authors declare no conflict of interest.

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