

Green Packaging: Carrageenan with Clove Oil Nanoencapsulation

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Abstract— The global urgency surrounding plastic waste-induced pollution has ignited significant interest in the biodegradable packaging sector, leading to a dynamic and expanding realm of biodegradable research. Nonetheless, effectively incorporating active agents into packaging materials faces hurdles related to stability and durability. This study takes a momentous leap in this context by shedding light on the transformative potential of integrating clove oil in macro and nanoencapsulation forms into carrageenan-glycerol matrices. A highlight emerges in the remarkable enhancement achieved through nanoencapsulation—a technique that substantially bolsters packaging performance. This improvement is evident in a noteworthy reduction of water vapor transmission, plummeting from an initial 11.011 to merely 6.903 g.mm m⁻² d⁻¹. This nearly fifty percent reduction underscores the efficacy of nanoencapsulation in enhancing packaging attributes. Concurrently, the study delves into the nuances of the carrageenan-clove oil composition, unveiling its profound influence on packaging strength, by systematically increasing the carrageenan-clove oil ratio, packaging strength triples, soaring from an initial 0.421 to an impressive 1.434 N/mm. Concerns of heightened brittleness due to increased carrageenan content are firmly dispelled, substantiated by consistent tensile stress values. In summary, this comprehensive investigation advances the core attributes of biodegradable packaging and presents a strategic and adept approach to address the intricate sustainability issues. Through adept manipulation of clove oil integration, the study pioneers the evolution of biodegradable packaging materials, paving the way for a more sustainable and environmentally conscious future.

Keywords— Biodegradable packaging; carrageenan; clove oil; glycerol; nanoencapsulation.

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I. INTRODUCTION

Plastic packaging has become an integral part of modern life, but it presents a significant environmental concern as it contributes to soil and water pollution [1]. Addressing this issue is crucial to minimize the environmental impact. Previous efforts have explored substituting plastic with starch-based materials, which can decompose naturally with the assistance of bacteria or fungi [2]. However, these substitutions have not entirely resolved the pollution problem since some petroleum-based chemicals remain, leading to harmful microplastics that threaten ecosystems. To overcome this, the development of non-plastic packaging with properties comparable to, or even superior to, plastic-based packaging is imperative.

Hydrocolloids are currently employed as stabilizing agents in various food and non-food formulations. For instance, carrageenan, sourced from *Kappaphycus alvarezii* and *Kappaphycus striatum algae*, has been used to produce kitchen utensils, hydrogels for controlled fertilizer mixtures,

and wound healing media. Consequently, carrageenan demonstrates potential as a fundamental material for environmentally friendly packaging. In earlier studies involving carrageenan, Laksono et al. [3] utilized it as a fertilizer carrier, implying its ability to biodegrade within the soil. This finding is supported by the research of Farhan and Hani [4], who used carrageenan as an active packaging to wrap chicken breasts. The packaging extended the shelf life of the chicken breasts and inhibited the growth of bacteria. Apart from carrageenan, alginate has also been used as the main ingredient in forming active edible films. These films have been shown to have antimicrobial properties and can also be used to control the moisture content of food products [5].

The characteristics of packaging materials should be carefully considered, particularly for shopping bags that require high strength and food packaging that necessitates factors such as water vapor transmission and antimicrobial properties [6]. Achieving desirable results in these aspects involves incorporating active ingredients into the packaging

formulation, making it comparable to conventional plastic packaging.

An antibacterial agent is an essential requirement for food packaging, and essential oils derived from lemongrass or cloves are potential candidates due to their high antioxidant value [7]. Additionally, by incorporating hydrophobic materials, water vapor transmission can be effectively reduced, ensuring the preservation of packaged food. This study aims to develop a hydrocolloid-based packaging formulation, employing carrageenan cross-linked with glycerol to create a thin, elastic layer suitable for wrapping food. The resulting packaging will exhibit reduced water vapor transmission and bacterial growth.

One of the extensively investigated ingredients is clove oil. Various research endeavors have focused on its potential applications. Noteworthy studies involving nanoencapsulated clove oil have contributed to its prominence. Sattary et al. (2020) have explored its utilization as a value-enhancing entity within the industrial domain [8]. Similarly, investigations by Abdelkhalek et al. [9] and Abaza et al. [10] have underscored its significance within the realm of health, particularly in terms of its antimicrobial and antioxidant properties. Furthermore, an intriguing dimension emerges through its incorporation as a supplementary component in preserving packaged food products [11]. This collective body of research highlights the multifaceted utility of clove oil and its nanoencapsulated variants across diverse sectors.

By employing carrageenan-based biodegradable packaging enriched with clove oil nanoencapsulation, this study seeks to contribute to sustainable packaging solutions that minimize environmental impact while meeting the functional demands of conventional plastic packaging. Such advancements can potentially revolutionize the packaging industry, promoting eco-friendly practices and ensuring a healthier ecosystem for future generations.

II. MATERIALS AND METHOD

A. Tools and Materials

The biodegradable packaging manufacturing process undertaken in this research necessitated the utilization of two primary components: carrageenan and glycerol, serving as fillers and stabilizers, and the active constituent, namely clove oil. The carrageenan employed in this study was sourced from PT Indoflora Ciptamandiri, while the clove oil utilized was procured from PT Scent Indonesia. In the realm of nanoencapsulation, the synthesis of clove oil involved the incorporation of several emulsifiers, specifically Tween 80 and Span 80. The process equipment includes a water bath, a desiccator, an oven, and glassware. Furthermore, analytical tools such as a Universal Testing Machine are utilized to assess the mechanical properties of the edible films.

B. Formulation

The samples were prepared by systematically varying the solid content, which involved combinations of carrageenan and glycerol and adjusting the concentration of the active ingredients. Three types of samples were created: the first group without any active ingredient, the second group containing the active ingredient in the form of clove oil, and the third group enriched with the active ingredient in clove oil

encapsulation. The composition of each sample can be found in Table 1.

TABLE I
VARIATIONS IN THE COMPOSITION OF PACKAGED EDIBLE SAMPLES

Materials	Sample composition (%)			
	I	II	III	IV
Carrageenan	2,5%	2,5%	2,5%	2,5%
Glycerol	3%	3%	3%	3%
Clove Oil	0%	0,43%	0,5%	1,1%*
Span 80	0%	0%	0,5%	0%
Tween 80	0%	0%	0,5%	0%
Water	94.50%	94.07%	93%	93.4%

*Sample IV using clove oil nanoencapsulation, while for samples II and III using clove oil extract.

Through this controlled sample preparation, the study aims to explore the effects of different formulations on the properties of the biodegradable edible films. The results will provide valuable insights into the optimal combination of ingredients for producing environmentally friendly packaging with enhanced functional characteristics.

C. Synthesis of Clove Oil Nanoencapsulation

The nanoemulsification process of clove oil eugenol was conducted using the Emulsion Phase Inversion Composition (EPIC) method. This method is a two-step process that involves the formation of a primary emulsion followed by the inversion of the emulsion phase [12]. In the first step, clove oil eugenol (the oil phase) was mixed with a nonionic surfactant (Tween-80) at room temperature (30°C) and 400 rpm. The oil-to-emulsifier ratio was 1:5, forming a primary emulsion with large oil droplets. In the second step, the primary emulsion was sonicated (using JP Selecta equipment) with power generated at 100 watts for 1 hour. The sonication process caused the oil droplets in the primary emulsion to invert, forming a nanoemulsion with much smaller oil droplets. The total processing time for producing the clove oil eugenol nanoemulsion was approximately 1.5 hours. The average droplet size of the nanoemulsion was 43 nm, as determined by dynamic light scattering. The nanoemulsion was stable for at least 6 months, as determined by measuring the droplet size. The nanoemulsion was also found to be biocompatible and non-toxic. This method is based on research by Singh et al. [13].

D. Particle Size Analyzer

Particle size measurements used the Horiba SZ-100 Particle Size Analyzer (PSA) with the laser diffraction (LAS) method.

E. Biodegradable Film Production

After the carrageenan-glycerol solution was uniformly dissolved, the solution was heated to 75°C and held for 10 minutes so that the carrageenan gelatinization process occurred. The temperature is then lowered to 60°C, the active ingredient of clove oil is added and stirred until homogeneous, and the temperature is held until all the air bubbles are released. After the carrageenan-glycerol solution was uniformly dissolved, it was heated to 75°C and held for 10 minutes so that the carrageenan gelatinization process occurred. The temperature was then lowered to 60 °C, and the active ingredient of clove oil was added and stirred until

homogeneous. The temperature was held until all the air bubbles were released. At 60°C, the solution was molded and dried under ambient conditions (24°C with 70% humidity) for 3 days. Fabricating this biodegradable packaging involves implementing the biofilm formation method as proposed by Zhang et al. [14].

F. Mechanical Test

Mechanical tests are carried out to measure tensile strength and tensile stress. Testing was carried out using a Universal Testing Machine. Before testing, biodegradable plastic film sheets measuring 6.4 x 0.6 cm underwent conditioning at 50% relative humidity (RH) for 48 hours. The Instron machine was configured with an initial grip separation of 50 mm, a crosshead speed of 50 mm/minute, and a 50 kg load cell. The plastic film was prepared using the ASTM-D638 standard [15].

G. Water vapor Transmission Rate (WVTR)

The WVTR of the film was assessed using the dish method E96. A cup containing silica gel as a desiccant was sealed with the film and placed inside a desiccator with controlled relative humidity and temperature set at 90% RH and 38°C, respectively. The cup's dimensions were 9,6 cm (outer diameter) by 7,8 cm (inner diameter) and 2,0 cm in depth, providing an exposed film area of 50 cm². The relative humidity within the cup was consistently lower than the external environment, facilitating the measurement of water vapor transport with a precision of 0,001 g at hourly intervals. The slopes, representing the change in cup weight over time, were computed for duplicate tests. Subsequently, the WVTR was calculated using the following method.

$$\text{WVTR (g. mm m}^{-2} \text{ d}^{-1}) = \frac{\Delta W(\text{g}).X(\text{mm})}{A.\Delta t.(\text{m}^2.\text{d})} \quad (1)$$

where ΔW is weight of water absorbed, A is area of the film, Δt is time for weight change, and X is film thickness. This method of measuring water vapor transmission was carried out according to research by Rahmi et al. [16] and Athanasopoulou et al. [17].

III. RESULTS AND DISCUSSION

A. Particle size of clove oil nanoencapsulation

Particle size analysis of clove oil nanoencapsulation plays a pivotal role in comprehending the physical attributes and stability of the encapsulated architecture [18]. In this research, laser diffraction techniques were employed to ascertain the distribution of particle sizes, with the outcomes visualized in Figure 1. The resultant average Z value of 17.5 nm signifies that most clove oil nanoparticles exhibited a diameter closely approximating this measure. This nanoscale magnitude holds considerable promise, serving as evidence for the productive creation of nanoparticles. Such achievement holds significance across diverse sectors, including pharmaceuticals, cosmetics, and the food industry. The diminutive particle dimensions confer distinct advantages, facilitating heightened bioavailability, bolstered stability, and precise modulation of the controlled release mechanism for the encapsulated bioactive constituents [19].

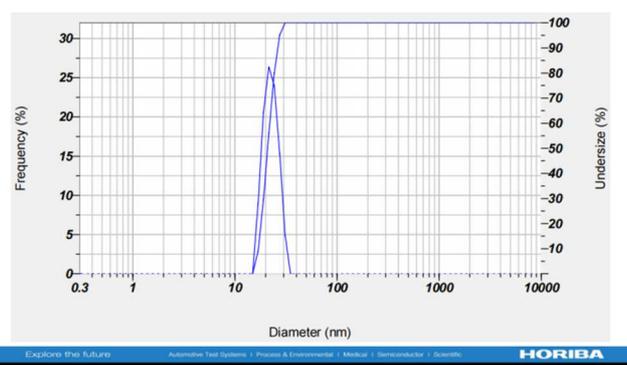


Fig. 1 Results of particle size analyzer measurements using the laser diffraction method

Simultaneously, the polydispersity index (PI) value of 0.637 indicates the breadth of the particle size distribution. Reduced PI values correlate with a narrower distribution, indicative of a relatively uniform assembly of particle sizes. In the context of this study, the PI value of 0.637 underlines the relatively narrow particle size distribution within the clove oil nanoparticles, substantiating a notable level of homogeneity in their size characteristics [20]. In general, a sample is considered to be nanosized if the Z-average is less than 100 nm. However, the PI value is also essential, as a high PI value can indicate that the sample is not homogeneous. A sample with a Z-average of 17.5 nm and a PI of 0.637 is considered a nanosized sample, but it is not perfectly homogeneous.

B. Interpretation of WVTR Test Results

The water vapor transmission rate is crucial to determine the film layer's permeability to water vapor. As emphasized in the introduction, the primary purpose of biodegradable packaging is for food applications, where it is essential to achieve a low WVTR value [21]. This low value signifies reduced water vapor penetration, which is critical as water can serve as a medium for bacterial growth [22]. The WVTR values obtained from the experimental analysis are presented in Table II.

TABLE II
WATER VAPOR TRANSMISSION RATE TEST RESULTS

Sample	WVTR value (g.mm m ⁻² d ⁻¹)
I	11.011
II	8.860
III	7.762
IV	6.903

This study aims to assess the WVTR of biodegradable edible films to ascertain their effectiveness in preserving and protecting packaged food items. The obtained WVTR data will contribute valuable insights to developing high-performance, environmentally friendly packaging solutions that align with food safety standards and sustainability principles.

The composition of the samples reveals a clear correlation between the addition of clove oil extract and the reduction in WVTR value. Including a hydrophobic material in the film layer decreases water activity, resulting in a diminished opportunity for water to permeate the film [23]. Interestingly, clove extract nanoencapsulation shows a remarkable

reduction in WVTR. This can be attributed to the nano-sized clove oil particles that efficiently spread, tightly cover, and fill the pores of the cross-linked carrageenan and glycerol network, creating a barrier that hinders water vapor penetration through the film.

Furthermore, WVTR values in samples II and III display differences, primarily due to the additional emulsifier used in sample III to incorporate the oil phase from the clove extract into the carrageenan solution. Consequently, sample III demonstrates a lower WVTR value as the cross-linked clove oil is more evenly distributed across the film's surface.

These findings shed light on the impact of incorporating clove oil extract and its nanoencapsulation in the formulation of biodegradable edible films. The observed reduction in WVTR emphasizes the potential of these films for practical food packaging applications, where minimized water vapor permeation is vital for ensuring food preservation and safety. Hydrophobic materials can be used as packaging coatings to reduce the value of WVTR. Understanding these phenomena paves the way for further advancements in eco-friendly packaging technologies with enhanced performance characteristics [24].

This mechanism closely resembles the behavior observed in hydrogel pores that act as oil absorbents in the petroleum industry. These pores are specifically employed to address oil leakage in aquatic environments. Figure 1 illustrates the gel structure formed as biodegradable packaging.

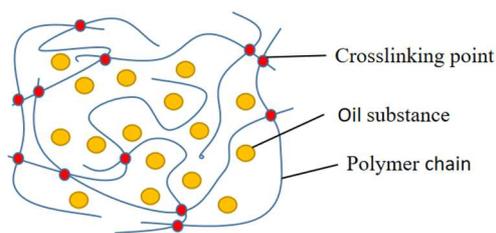


Fig. 2 Polymer bond structure with enrichment of the active ingredient in the form of oil

The depiction in Figure 2 illustrates the configuration of carrageenan polymer chains in conjunction with glycerol, giving rise to dynamic polymer chains capable of interconnecting with additional carrageenan polymers. Clove oil can ingress and find its place within the interconnections, driven by van der Waals forces [25]. These circumstances collectively create an environment that restricts the permeation of water vapor into the packaging's pores.

C. Interpretation of Mechanical Test Results

Tensile strength measures a sample's maximum strength before tearing, gauging the force needed for maximum pull across its surface. Raw materials concentration, type, and structural cohesion—intermolecular bonding— influence it. The results can be seen in Table III.

TABLE III
MECHANICAL TEST RESULTS

Sample	Tensile Strength (N/mm)	Tensile Stress (MPa)
I	0.421	10.772
II	0.793	11.709
III	0.669	8.909
IV	1.434	11.833

The analysis of the tensile strength data reveals a discernible influence arising from incorporating carrageenan-glycerol fillers. Furthermore, a notable disparity is observed among the samples denoted as I, II, and III, wherein an evident enhancement in tensile strength is evidenced. This augmentation underscores the constructive role of the active constituents in fortifying the inherent physical attributes of the biodegradable packaging material.

An intriguing phenomenon surfaces in the case of sample IV, characterized by a tensile strength value twice that of samples II and III. This compelling outcome underscores the consequential impact of nano-shaped active materials on the resultant tensile strength [26]. This underscores the multifaceted role of carrageenan in influencing the material's mechanical properties. Adding nano-encapsulated clove oil to biodegradable packaging can further improve their tensile strength. The nanoencapsulated clove oil is more evenly distributed throughout the material than the other samples. The nanoencapsulated clove oil is surrounded by a polymer layer, preventing it from migrating to the material's surface [27].

Carrageenan can form associations with glycerol and water, resulting in a material that displays pronounced temperature sensitivity, thereby inducing alterations in the inherent characteristics of the gel matrix [28]. The augmentation of constituent materials is poised to positively influence the mechanical attributes of the resultant gel product [29]. Carrageenan possesses the ability to establish connections with diverse starches. Glycerol, in this context, functions as a vital adhesive agent, fostering interlinkages amidst the polymer chains of the starch. This phenomenon is facilitated by providing thermal energy and agitation during the gelation process, culminating in the development of a robust gel structure [30].

Based on the attained results, it is evident that applying nanoencapsulation can indeed augment the robustness of biodegradable packaging. Baghi et al. [31], similarly highlighted the potential of nanoparticles in fortifying packaging strength, consequently extending its shelf life. On the other hand, in terms of tensile stress, negligible distinctions are discernible across nearly all samples, indicating a uniform strength among them when subjected to forces leading to fracture.

D. Effect of Nanoencapsulation of Clove Oil

This research is oriented towards developing a packaging solution capable of effectively impeding water vapor transmission while demonstrating resilience against bacterial agents. Clove essential oil is an active ingredient within the carrageenan gel network to achieve this objective. The outcomes from both Water Vapor Transmission Rate (WVTR) measurements and mechanical assessments unequivocally highlight the favorable impact stemming from the utilization of clove oil, particularly when nanoencapsulated. This enhancement is closely intertwined with the intricate gel structure, as depicted in Figure 2. Within this structure, oil constituents can exist in two states: unbound, signifying oil not integrated into the gel network, and bound, representing oil emulsified and subsequently assimilated into the gel network. Both these states synergistically augment the biodegradable attributes of the resulting packaging material.

Active ingredients in their macro form are more likely not to be absorbed and only coat the outer surface of the packaging. This can lead to decreased product longevity and increased water vapor transmission. Additionally, the gel structure may become more brittle over time due to the large number of cavities or pore sizes in the matrix.

Employing a nanoencapsulation system presents an advantageous technological approach. It offers benefits such as deterring undesired interactions, ensuring heightened stability, enhancing bioavailability, and manifesting potent antimicrobial and antioxidant activities during food preservation. As evidenced by Bahrami et al. [32], this integration of nanoencapsulation technology with packaging holds significant promise.

IV. CONCLUSION

Carrageenan-glycerol can be used as the basis for making biodegradable packaging. The combination of carrageenan-glycerol and clove oil nanoencapsulation significantly improved the properties of the biodegradable packaging, including the water vapor transmission value and the tensile strength. The nanoencapsulated clove oil was trapped in the cross-links formed by carrageenan-glycerol, which resulted in better stability and distribution of the clove oil in the packaging. The results of this study suggest that carrageenan-glycerol with clove oil nanoencapsulation is a promising material for biodegradable packaging. The packaging has good mechanical properties and retains water vapor transmission value. The nanoencapsulated clove oil also has potential antimicrobial properties, which could make the packaging more resistant to microbial spoilage. Further studies are needed to investigate carrageenan-glycerol's long-term stability and antimicrobial properties with clove oil nanoencapsulation. However, the results of this study suggest that this material has potential for use in biodegradable food packaging.

NOMENCLATURE

Cumulant Operations	
Z-Average	17.5 nm
Polydispersity Index	0.637

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