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PREPARATION AND CHARACTERIZATION OF K-CARRAGEENAN BASED GREEN CAPSULE FILMS ENHANCED WITH PECTIN

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ABSTRACT

The current study aimed to development green capsule films made from k-carrageenan (CAR) enhanced with pectin (PEC). Most capsules are constructed from gelatin animal, although localization capsule is effective, there are challenges in their application such as brittleness and safety concerns. Carrageenan from seaweed sustainable alternative but exhibits low mechanical stability. Addition of pectin for biocompatibility and ability to improve material properties. The researchers aimed to strengthen the CAR matrix. The CAR/PEC capsule are prepared dan analyzed for mechanical properties, FTIR spectroscopy, surface morphology using SEM, moisture content, and disintegration time. Based on this research, can highlighted that adding pectin improved the mechanical and functional properties of the capsule films, making them a viable, eco-friendly as alternative to localization gelatin-based capsules

KEYWORDS: k-carrageenan (CAR), pectin (PEC), capsule film

1. INTRODUCTION

Capsules used as pharmaceutical excipients to encapsulate pharmaceutical active ingredients in a shell that is elegant, odorless, tasteless, and easy to ingest [1]. They provide advantages such as enhanced bioavailability, improved drug stability, and efficient localization and release of the active ingredients [2]. Most capsules are constructed from gelatin sourced from animals, which is characterized by its superior film-forming properties, high production efficiency, and excellent overall [3]. However, gelatin-based capsules exhibit several notable limitations that restrict their broader application. For instance, they are susceptible to cross-linking reactions (both chemical and molecular bonds), become brittle in low humidity conditions, soften at high temperatures, and certain components (such as heavy metals,

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preservatives, and bacteriostats) can often exceed permissible limits [4]. Additionally, being an animal-derived material, gelatin carries the risk of transmitting viruses (such as "mad cow disease"), and it poses ethical concerns for specific groups, such as vegetarians and individuals with certain religious beliefs [5]. Consequently, the research and development of eco-friendly, biodegradable, non-gelatin capsules hold significant importance.

Carrageenan has emerged as a promising alternative raw material for capsule shell production, offering a viable substitute for gelatin. Extracted from seaweed, it is abundant in the extensive tropical waters of Indonesia, making it a valuable and sustainable resource [6]. Currently, carrageenan is widely utilized across various industries, including food, pharmaceuticals, nutraceuticals, cosmeceuticals, medical, and others, primarily as a thickening agent and filler [7]. However, carrageenan was employed as the primary raw material for capsule formulation. Despite its potential, carrageenan tends to become brittle during the drying process due to its low mechanical stability and the formation of double helices within its matrix [8].

The addition of pectin offers dual advantages, specifically improving both the mechanical and functional properties of materials [9]. Pectin is a high molecular weight natural polysaccharide that is biocompatible, non-toxic, and anionic, derived from plant cell walls. It is acknowledged as a functional ingredient, gel-forming agent, and stabilizer within the food industry. Due to its unique characteristics, pectin emerges as an appealing new biopolymer with potential applications in the pharmaceutical and healthcare sectors [10]. Its favorable drug delivery capabilities stem from its chemical composition and the functional groups present on its surface [11]. Pectin is predominantly found in the cell walls of green plants, especially in fruits such as apples, lemons, and oranges [12].

In this research, capsule films were formulated by combining k-carrageenan as the matrix and gelling agent, with pectin serving as the reinforcing component and polyethylene glycol functioning as the plasticizer. The study conducted a detailed analysis of mechanical properties, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), loss on drying, and viscosity, highlighting the significant potential of the k-carrageenan/pectin (CAR/PEC) system in the development of green capsule films.

2. MATERIALS AND METHODS

2.1 Materials

Pectin was supplied by PT Reinaldoi (Surabaya, Indonesia) and the average particle size of pectin was 70 nm. Kappa-carrageenan (molar mass 401.32 g/mol with CAS number 11114- 20-8 SIGMA-Aldrich, USA). Polyethylene glycol (PEG) 400 and HCl were supplied by PT Indrasari (Semarang, Indonesia). The

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stainless-steel mold for the capsule shell (cylindrical, 9.55 mm in diameter) was made by ourselves.

2.2 Preparation of the capsule-film and capsule

2.2.1 Preparation of the CAR/PEC gel solution

The gel solution was prepared with 5 gr CAR and different proportions of PEC (based on 0 gr, 1 gr, 3 gr, 5 gr and 7 gr). The polyethylene glycol (PEG) 400 was used as plasticizer and gelling agent, respectively. Different proportions of CAR/PEC were added to a beaker of distilled water and then slowly added 4 mL PEG 400. The mixed solution was stirred at a high speed (530 rpm) at 70 °C for 20 min, and then stirred at a low speed (200 rpm) for 20 min to obtain the transparent gel solution.

2.2.2 Preparation of the CAR/PEC capsule-film

Solution casting method was used to prepare capsule-film with the gel solution obtained in Section 2.2.1. The mixed solution was put directly onto the tray and dried at 55°C until the CAR/PEC capsule-film with different ratios of PEC formed. CAR/PEC-0, CAR/PEC-1, CAR/PEC-3, CAR/PEC-5, and CAR/PEC-7 represented different k-carrageenan-based capsule-film with 0 gr, 1 gr, 3 gr, 5 gr and 7 gr of pectin, respectively. The average thickness of the CAR/PEC capsule-film was 0.20 mm.

2.2.3 Preparation of the CAR/PEC capsule

The gel solution obtained in Section 2.2.1. was stirred at a speed of 400 rpm at 70 °C for 120 min. Then the gel solution was cultured at 50 °C for 30 min. Capsules were prepared by dipping stainless steel mold into the gel solutions for 6–8 s. When the mold was withdrawn, a film of gelled solution remained on the mold. The coated mold was then dried at 55°C. The drying time depended on the rigidity of the capsule. The dried CAR/PEC capsules were then stripped and cut into sizes. The average thickness of CAR/PEC capsule was 0.18 mm.

2.3 Tensile strength

The tensile strength of the CAR/PEC capsule-film sample was tested by the texture analyzer Brookfield CT 03 4500. The tensile strength (TS, MPa) was calculated by the following equation:

$$Tensile\ \ Strength\ \ (MPa) = \frac{F}{A} \qquad (1)$$

Where F is the maximum load (N); and A indicates the cross-sectional area (mm^2).

2.4 Moisture content

The sample was cut, then weighed as much as 1 gram, after which it was put into a cup. Next, the cup

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containing the sample was heated at 110°C for 3 hours in an oven. The sample was cooled in a desiccator for 30 minutes and then weighed, this last step was repeated until the weight was constant. Loss on drying was calculated by the following equation:

$$Moisture\ \ Content\ \ (\%) = \frac{W_1 - W_2}{W_1} \times 100\% \qquad (2)$$

Where W_1 is total weight before drying, and W_2 is total weight after drying. Each sample was measured in triplicate.

2.5 Disintegration time

The disintegration time of the CAR/PEC capsule in purified water and hydrochloric acid (HCl) was determined by using TEQ disintegration tester.

2.6 Fourier transform infrared (FTIR) spectroscopy

The CAR/PEC capsule was analyzed for the chemical groups contained by Fourier-Transform Infrared Spectroscopy (FTIR) Perkin Elmer Spectrum IR 10.6.1 spectrophotometer (Perkin Elmer Inc., US) with a spectrum of 4000-400 cm^{-1}.

3. RESULTS AND DISCUSSIONS

3.1 Tensile strength

The effect of different pectin concentration from 0% to 7% on tensile strength of CAR/PEC-based capsule-film is shown in Figure 1. The results indicated that pectin had obvious reinforcing effect on the CAR/PEC-based capsule-film. It can be seen that the tensile strength of the capsule-film increased first and then decreased with the increase of pectin content. When the pectin content was 5% wt, the tensile strength of CAR/PEC-based capsule-film increased by 118.14% and reached the maximum of 4.45 MPa. However, when the pectin content was 7% wt, the tensile strength of the CAR/PEC-based capsule-film decreased slightly. Tensile strength can provide a good indication for the performance of CAR/PEC-based capsule-film capsule.

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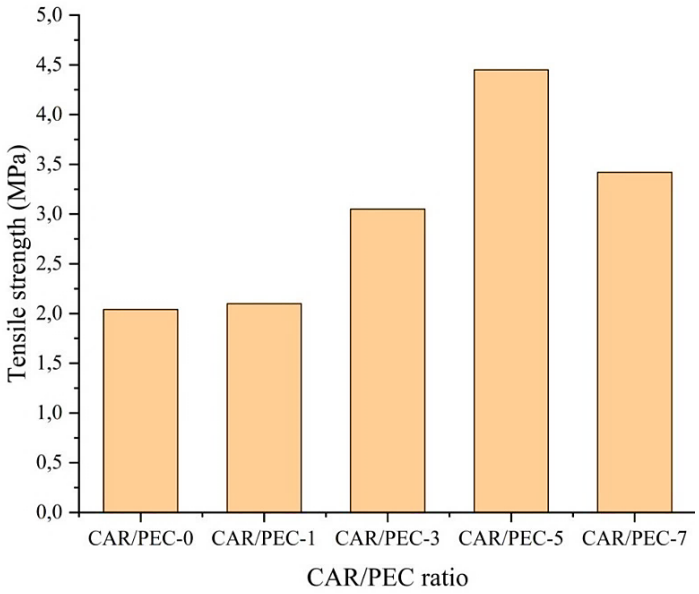

Figure 1: Effect of pectin content on the tensile strength of capsule-film

Tensile strength can provide a good indication for the performance of CAR/PEC-based capsule-film capsule. This is consistent with the previous study [13], which found that as pectin concentration increased, the film became stronger and had a higher capacity to extend before breaking. The tensile strength was increased because of the increased interaction among polymers inside the matrix of films leading to more compact film structure. Tensile strength is significantly affected by intra and inter molecular network bonds, concentration of components, film constituents, and preparation method. The reason for increase in tensile strength may be the increased intermolecular interactions causing lesser mobility of molecules inside film matrix thus creating less space for chains of polymer to slide [14]. However, when the PEC content was at 7% weight, the tensile strength of the CAR/PEC capsule film experienced a slight decrease. This could be attributed to the self-aggregation of the added PEC, which led to an uneven distribution of tension within the capsule film [15].

3.1 Moisture content

In the process of capsule production, it is very important to control the moisture content of capsules. The moisture content of capsules directly affects the quality, stability, disintegration time and hardness, etc. The results of the moisture content calculation for the CAR/PEC capsules are shown in Table 1.

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Table 1: Moisture content of the CAR/PEC capsule

Capsules with Different PEC Contents	*Moisture Content (%)*
CAR/PEC-0	11.25
CAR/PEC-1	12.93
CAR/PEC-3	13.87
CAR/PEC-5	14.30
CAR/PEC-7	13.02

Table 1. shows the moisture content of the CAR/PEC capsules with different PEC contents. It can be seen that there is no significant difference between the developed CAR/PEC capsules. The results showed a tendency for the moisture content to increase with increasing added pectin quantity. The United States Pharmacopoeia stipulates that the moisture content of capsules should be between 10% - 15%. The final moisture content was ranging from 11,25-14,30%, which is in line with the provisions of the United States Pharmacopoeia.

Yap et al. (2022) [16] reported that increasing the concentration of pectin can improve the water-absorbing properties of the gel. This suggests that the physicochemical properties of the pectin itself may play a role in determining the moisture content in the final product. Pectin is a hydrophilic (water-attracting) polysaccharide, and as more of it is incorporated into the formulation, it binds to more water molecules. This increase in water binding capacity raises the moisture content in the capsule. Pectin also increases the viscosity of a solution because it forms a network structure in water, which thickens the solution. This happens because pectin molecules have long, flexible chains that interact with water molecules and each other, creating a more structured, gel-like system. As the concentration of pectin increases, these interactions become stronger, resulting in higher resistance to flow, which is perceived as an increase in viscosity [17]. As viscosity rises, water moves more slowly through the system, which allows the capsule to retain more moisture. Too high concentration of pectin can cause the formulation to become too dense or form a gel-like structure. This dense network of pectin molecules makes it harder for additional water to be absorbed, as there are fewer spaces available within the structure to hold the water [18]. At this point, the moisture content can actually decrease because the excess pectin molecules trap water less effectively. Therefore, finding the right balance in pectin concentration is essential to optimize both moisture content and viscosity in formulations. The results of this study show a similar trend to the research conducted by Rana et al. (2024) [19], who performed pectin in hydrogel. In that study, showed that there is an optimal concentration range for pectin to maximize moisture retention without reducing moisture content.

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3.3 Disintegration time

The disintegration time plays an important role for the drug in the capsule before it is absorbed by the body. The capsule must completely disintegrate before the effect of the drug is exerted, which is necessary and crucial for the development of rapidly disintegrating capsules. human health. Table 2 shows the disintegration time of different capsules in water and HCl.

Table 2: Disintegration time of the CAR/PEC capsule

Capsule Style	*Completely disintegration time*	
	In Water	*In HCl*
CAR/PEC-0	11′42″	03′29″
CAR/PEC-1	13′27″	04′37″
CAR/PEC-3	13′09″	04′42″
CAR/PEC-5	12′48″	04′46″
CAR/PEC-7	13′10″	05′10″

According to Table 2. the disintegration time of CAR/PEC capsule in HCl was faster than the disintegration time in water. The results of the analysis also showed that the disintegration time of capsules containing different concentrations of PEC was not very different, the average disintegration time was 4-5 minutes. The fastest disintegration time of CAR/PEC capsules is found in capsules with 0 g pectin concentration with a disintegration time of 3 minutes 29 seconds, while the longest disintegration time is found in capsules with 5 g pectin with a disintegration time of 5 minutes 10 seconds.

Pectin improves the surface structure of CAR/PEC capsules by forming a cohesive gel matrix that strengthens the capsule walls [20]. This structural improvement increases viscosity as the denser network created by pectin as polymer slows the diffusion of dissolution media [21]. Higher viscosity results in longer disintegration times as the gel barrier resists rapid degradation]. The results are consistent with viscosity tests where capsules with higher PEC concentrations showed delayed disintegration times, confirming that increased viscosity correlates with slower capsule dissolution [22].

The results of this study show a similar trend to the research conducted by Chen et al. (2021) [15], who added cellulose nanocrystals (CNC) to a mixture of green nanostarch capsules (CNS). In that study, the addition of CNC into the CNS can effectively improve the surface structure of the capsule, and the disintegration time was relatively longer than that of the capsule without CNC.

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3.4 Fourier transform infrared (FTIR) spectroscopy

To determine the effects of pectin addition process on the functional groups of capsules made from k-carrageenan, the FTIR test results are presented in Figure 2.

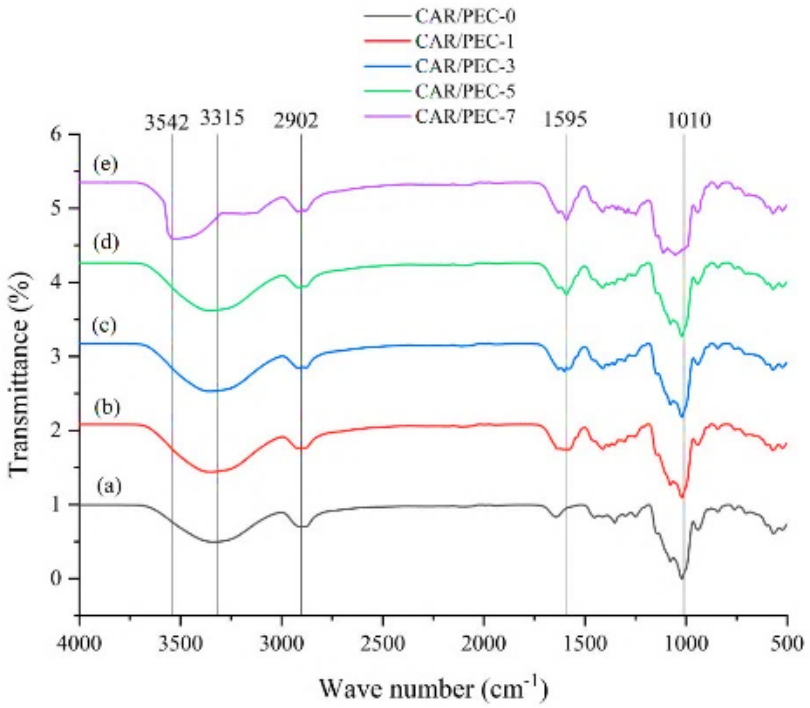

Figure 2: FTIR spectra of (a) CAR/PEC-0, (b) CAR/PEC-1, (c) CAR/PEC-3, (d) CAR/PEC-5, (e) CAR/PEC-7

Based on Figure (a), the wavelength of 3542 cm^{-1} is due to the stretching vibration of the hydroxyl (–OH) group, which is characteristic of the polysaccharides found in both kappa carrageenan and pectin. The intensity of this peak increases with the addition of pectin, indicating a stronger hydrogen bonding network in the material [23]. At a wavelength of 3315 cm^{-1}, stretching of the hydroxyl group is also observed, which suggests the presence of intermolecular hydrogen bonds between the pectin and kappa carrageenan molecules. At a wavelength of 2902 cm^{-1}, a C–H stretching band is observed, indicating the presence of aliphatic chains within the kappa carrageenan and pectin structures. This peak remains consistent across all samples, indicating that the aliphatic nature of the polysaccharides does not change significantly with the concentration of pectin [24]. In Figure (b), the peak at 1595 cm^{-1} corresponds to the stretching vibration of the carbonyl (C=O) group from the carboxylate anion, which is primarily found in pectin. The intensity of this peak increases with the concentration of pectin, showing that the carboxyl groups from pectin are becoming more dominant in the composite material. At 1010 cm^{-1}, the C–O–C glycosidic bond is observed, which is a key structural feature of both kappa carrageenan and pectin. The intensity of this

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peak also increases with the addition of more pectin, suggesting that the glycosidic linkages in pectin are contributing to the overall structure of the film [23]. Figures (c), (d), and (e) show that the concentration of pectin directly influences the intensity of the characteristic peaks associated with both the hydroxyl and carbonyl groups, indicating enhanced hydrogen bonding and stronger interactions within the matrix. These changes in the FTIR spectra suggest that higher pectin concentrations lead to more structured and stable interactions within the kappa carrageenan matrix, possibly improving the mechanical and barrier properties of the film [24]. It can be concluded that the addition of pectin significantly influences the functional group interactions within the kappa carrageenan-based capsule film. The hydrogen bonding between hydroxyl groups and the interaction of carboxylate groups increase with higher pectin concentrations, leading to a more stable and organized structure. These findings suggest that adjusting the pectin concentration can optimize the mechanical properties of the film for potential applications in capsule production.

4. CONCLUSIONS

Pectin added to k-carrageenan have the potential to be innovative alternative in producing environmentally friendly and biodegradable capsule films. In this study shows that the addition of pectin to k-carrageenan can enhance the viscosity of the gel solution, mechanical strength, moisture retention, disintegration efficiency, influence the functional group interactions, and increase electrostatic repulsion. The most optimal results in this study were achieved with a 5% pectin content. Overall, according this study it shows that the CAR/PEC blend has effective potential to be developed as a gelatin substitute, particularly in the pharmaceutical industry for a more environmentally friendly approach.

Declaration of Competing Interest

The authors declare that they no known competing financial or personal interests that could have to influence or compromising the integrity of the work reported in this paper.

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