

Mechanical and Barrier Properties of Semi Refined Kappa Carrageenan-based Composite Edible Film and Its Application on Minimally Processed Chicken Breast Fillet

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Abstract. Kappa-carrageenan (KC) is one of the most interesting biopolymers that is composed of a linear chain of sulfated galactans and extracted from red seaweed, *Kappaphycus alvarezii*. It shows good potential for development as a source of biodegradable or edible films. However, KC films do not have good water vapor barrier properties, as they are intrinsically hydrophilic. Palmitic acid (PA) as hydrophobic material was incorporated into semi-refined kappa-carrageenan (SRKC) edible films in order to improve water vapor barrier properties. In this study, composite films based on SRKC incorporating PA were prepared and their applications on minimally processed chicken breast fillet were evaluated. Composite SRKC-based films with varying concentrations of PA (5%, 10%, and 15% w/w) were obtained by a solvent casting method. Their mechanical and barrier properties were investigated. Results showed that the incorporation of PA in films caused an increase in thickness, but decrease in water vapor transmission rate (WVTR) as the concentration of PA increased (from 5% to 15% w/w). Composite SRKC-based edible film incorporating 15% w/w of PA presented better water vapor barrier properties as compared to other films with 5% and 10% w/w PA incorporation. Thus, formulation containing 15% w/w PA was used as a wrapping material for film application on minimally processed chicken breast fillet. The application results showed that the incorporation of PA in film caused an effect ($p < 0.05$) on preventing of weight loss significantly compare to control (non-wrapping), however it did not significantly ($p > 0.05$) change the color of minimally processed chicken breast fillet.

1. Introduction

Global demand for meat is expected to increase by 44% by more than 400 million tonnes by 2030 with demand for poultry by 39% [1]. While increasing consumer demand for high food quality, fresh, nutritious, and easy to prepare caused minimally processed food is more preferred, including fresh chicken meat. However, the main obstacle for this commercial product is short shelf life due to high nutritional content and moisture content in fresh chicken meat resulting in the rapid growth of decaying microorganisms and pathogens. Chicken breast meat can be stored at a temperature not exceeding 4°C to preserve its good condition for 4-5 days. While the broiler chicken pieces stored at a temperature of 28°C has a shelf life for 8 hours [2]. Edible films and/or coatings can improve the quality of poultry meat, by reducing the loss of moisture content on the meat surface, inhibiting discoloration due to the combination of lipid oxidation and myoglobin oxidation, and inhibiting the activity of decaying microorganisms and pathogens [3]. Therefore, the application of edible films



and/or coatings on chicken breast fillet is expected to be alternative natural ingredients for packaging that can prolong the shelf life of fresh and/or minimally processed chicken breast fillet, so that consumer demand for high quality fresh chicken breast fillets can be fulfilled. Different structural materials have been used for the development of edible films and/or coatings, such as proteins, lipids, and polysaccharides. Film-forming materials can be utilized individually or as mixed composite blends. Proteins and polysaccharides used for their mechanical and structural properties, while hydrophobic substances (lipids (fatty acids), essential oils, and emulsifiers) used for providing good moisture barrier properties [4,5,6,7].

Kappa carrageenan is water-soluble polymer with a linear chain of partially sulphated galactans, which present high potentiality as film-forming material. Shorter and cheaper extraction method of red alga, *Kappaphycus alvarezii*, could be resulted in semi-refined kappa carrageenan (SRKC) of which a renewable resource and commercially available at a reasonable cost. Carrageenan-based edible films are brittle, thus a plasticizer to improve the mechanical properties is needed [6,8,9]. Sorbitol was employed as plasticizer in this work due to its several advantages such as retaining moisture and demonstrating stable performance at high temperature [10]. Moreover, hydrophobic materials such as fatty acids could be used to enhance the barrier properties of this film. Thus, this present study deals with the formulation and characterization of a semi-refined kappa carrageenan-based composite edible film incorporated with palmitic acid and evaluation its application on minimally processed chicken breast fillet.

2. Experimental

Semi-refined Kappa-carrageenan, as the main component of the film matrix, was purchased from Galic Artabahari, Co., Ltd. (Cikarang Barat, Indonesia). Sorbitol (plasticizer) and palmitic acid (PA) were purchased from Brata Chemical (Surakarta, Indonesia) and Chemix Pratama (Yogyakarta, Indonesia), respectively. Zein was obtained from Laboratory of Food Chemistry and Biochemistry, Gadjah Mada University (Yogyakarta, Indonesia). Minimally processed chicken breast fillet was purchased from local market 'Pasar Demangan' (Yogyakarta, Indonesia). Aquadest was used for all sample preparations and all other chemicals used were in analytical grade.

Composite Semi-refined Kappa Carrageenan (SRKC)-based edible films were produced as described in Manuhara *et al.* with modification [11] and the SRKC concentration was selected based on the best properties of SRKC film as described in our previous study [9]. In this study, two groups of solutions were prepared. The first group, zein solution of 5% (w/w semi-refined kappa carrageenan) was prepared by dissolved in 10 ml of 96% ethanol, and stirred with a magnetic stirrer for 10 minutes at room temperature ($28 \pm 2^\circ\text{C}$). Palmitic acid at various concentrations (5, 10, and 15%, w/w dry SRKC) was then added into zein solution, stirred with a magnetic stirrer for 10 minutes until it became completely homogenous (Solution A). The other group was the preparation of the main solution. It was prepared by dispersing 2 g of SRKC in aquadest (90 ml) and stirred under continuous stirring until dissolved and reached the temperature of 60°C . It was maintained for 10 minutes. Sorbitol liquid (1% v/v) was then added as the plasticizer and the Solution A were also poured into a main film solution. The mixture was then heated to 80°C and the temperature of mixture was then maintained at 80°C for 5 minutes on a hot plate with constant stirring to obtain a homogeneous solution. The film-forming emulsions were left for several minutes and stirred manually with stainless steel spatula to naturally remove dissolved air bubbles produced during stirring. The film-forming emulsions were then casted by pouring onto a casting plate (W x L x H: $15 \times 23 \times 2$ cm) evenly and immediately to avoid the solution to turn into a gel. The plates were dried at 60°C in drying oven for 8 hours. The dried film were carefully peeled off from the plates and dried at room temperature ($28 \pm 2^\circ\text{C}$) for 24 hours with 50% RH condition before analysis [6,9,11].

Mechanical (thickness, tensile strength (TS), and elongation at break (EAB)), and barrier properties of composite SRKC-based edible films were analyzed by methods as described in our previous work [6,9]. The water vapor transmission rate (WVTR) of the film specimens was measured according to a modified ASTM E96/E96M-05 method 1997 [12], and its analysis was conducted as

mentioned in our previous work [6,9]. Weight loss for film application by wrapping was conducted by a method as described in our previous work and color was analyzed by a method of Andarwulan et al. [13]. A total of 3 samples were examined for each film type, thus, each film sample consisted of two replicate measurements. The experiments of factorial with a completely randomized design were used. The various concentrations of palmitic acid were the independent variable. All data were statistically evaluated by analysis of variance (ANOVA) procedure using SPSS software (version 16.0 for Windows software; SPSS Inc., Chicago, USA). Duncan's multiple range tests were used to compare the difference among the mean values for the film properties. Differences between the mean values were considered significant at $p < 0.05$.

3. Results and Discussion

Table 1. Mechanical and Barrier Properties of Composite Semi-Refined Kappa Carrageenan (SRKC)-based Films

Palmitic Acid (% w/w)	Thickness (mm)	TS (MPa)	EAB (%)	WVTR (g/h.m ²)
5	0.063 ^a ±0.00	16.82 ^a ±2.15	9.89 ^a ±4.54	8.93 ^a ±1.89
10	0.067 ^a ±0.01	13.84 ^a ±2.90	9.86 ^a ±3.47	8.26 ^a ±2.03
15	0.070 ^a ±0.00	14.64 ^a ±2.90	9.88 ^a ±2.77	6.51 ^a ±1.58

TS: Tensile strength; EAB: Elongation-at-break; WVTR: Water Vapor Transmission Rate

In the present study, the thickness of composite Semi-refined Kappa Carrageenan (SRKC)- based films were ranged from 0.063 to 0.070 mm, and as a comparison (0% PA concentration), the thickness was 0.053 mm [11]. The lowest thickness of composite SRKC-films were obtained at the 5% w/w PA concentration, whereas the highest thickness was obtained at the 15% w/w PA concentration (Table 1.), however they did not show significantly differences ($p > 0.05$) on thickness parameter. Tensile strength (TS) and elongation-at-break (EAB) are included in mechanical properties of the composite SRKC-based films as shown in Table 1. TS indicate the maximum tensile stress that the film can sustain, while EAB is the maximum change in length of a test specimen before it breaks [14]. The low interval concentration (5%) of palmitic acid incorporation to the films did not give any significant effects to TS and EAB of composite SRKC-films. The percentage of TS and EAB were decreased and then increased. The decrease occurred at a 10% concentration of palmitic acid addition, and increased at a concentration of 15%. The TS of composite SRKC-films were ranged from 13.84 to 16.82 MPa, while EAB of composite SRKC-films were ranged from 9.86 to 9.89 %. As comparison (0% PA concentration), TS and EAB of composite SRKC-films were 21.14 MPa and 12.36% [11], respectively. Controlling mass transfer plays important role of food packaging for preservation of food. Hydrophobic compounds with their non-polar molecules can improve moisture migration, thus they could be employed as effective barriers [15]. The WVTR values of the composite SRKC films at 28±2°C and 50% RH are shown in Table 1. The addition of 15% w/w PA was the most effective in improving water barrier properties. However, no significant difference ($p > 0.05$) was observed in comparing to the films produced in this study. Interestingly, the decline in film WVTR is possibly to be associated with the film thickness. For instance, as the palmitic acid concentration increased, the thickness successively increased (Table 1), followed by a decrease in WVTR values (Table 1), probably due to the significant reduction of water mobility through the film. Formulation containing 15% w/w PA was then used as a wrapping material for film application on minimally processed chicken breast fillet.

Table 2. Weight Loss of Minimally Processed Chicken Breast Fillet

Treatment	Weight Loss (g/h)
Control (non-wrapping)	0.101 ^c ±0.002
Wrap plastic	0.003 ^a ±0.000
Composite SRKC-film	0.067 ^b ±0.002

Weight loss in food is generally associated with decreased moisture content. The use of film as a wrapping material of food is determined by the film's ability to reduce moisture loss in food. The weight loss analysis on minimally processed chicken breast fillet was conducted by weighing chicken breast fillets, either wrapped with composite SRKC film, wrapping with cling wrap, or non-wrapping (control) for 8 hours. Chicken breast fillet was placed on acrylic cup and stored in desiccator containing saturated silica gel (RH 0%). Weighing is done from hour 0 to hour 8 every 1 hour. During storage there was weight loss of chicken breast fillet. Table 2. shows that wrapping application by wrap plastic and composite SRKC film inhibited weight loss of minimally processed chicken breast fillet during treatment. It can be seen that control (non-wrapping) demonstrated the highest of weight loss (0.101 g/h), while chicken breast fillet wrapped by composite SRKC film 0.067 g / h and by plastic wrap 0.003 g/h. Thus, packaging of minimally processed chicken breast fillet in wrapping treatment with composite SRKC film can reduce the weight loss of chicken breast fillet by about 2 times and had a significant effect ($p < 0.05$) on reducing weight loss after 8 hours of storage compared to control (non-wrapping). While wrapping with wrap plastic able to minimize weight loss of chicken breast fillet approximately 30 times compared to control. Therefore, it may suggests that the composite SRKC film used in this study provides barrier properties against water vapor, i.e. by inhibiting the loss of water content in chicken breast fillets, but the wrap plastic provides the barrier properties better than composite SRKC film. This is because the wrap plastic is a plastic made of polyvinyl chloride, which is a flexible plastic that is obtained by the addition of plastic materials and has a smaller pore size (density) so as to withstand the rate of water vapor transmission [16].

Table 3. Color Analysis of Minimally Processed Chicken Breast Fillet

Treatment	L*	a*	b*	ΔE
Control (non-wrapping)	42.06 ^a ±1.59	3.26 ^a ±1.37	8.50 ^a ±1.82	-
Wrap plastic	44.20 ^a ±1.57	1.53 ^a ±0.87	6.10 ^a ±0.14	3.66
Composite SRKC-film	42.95 ^a ±1.27	3.28 ^a ±0.56	7.30 ^a ±0.98	1.50

The color of the meat is a visual parameter that is first seen by consumers as an indicator of freshness. It is related to pigment concentrations, especially myoglobin and hemoglobin. The color change of meat during storage is influenced by several factors, including the level of metmyoglobin accumulation. Metmyoglobin produces a brown color in the flesh thus lowering the freshness and the level of consumer acceptance of meat. The rate of metmyoglobin accumulation is related to intrinsic factors, such as pH and fiber types in animal muscle, and extrinsic factors, such as post-slaughter treatment and carcass chilling treatment. Environmental factors also affect meat color changes, such as temperature, oxygen, light, microbial growth, and meat storage packaging. The discoloration of meat during storage occurs as a combination of pigment oxidation and lipid oxidation in meat [17]. Moreover, biopolymer-based edible films used as packaging material of meat products can reduce water loss, minimize lipid oxidation, and prevent discoloration [18]. This color analysis aimed to determine the effect of wrapping treatment by composite SRKC film in maintaining the color of minimally processed chicken breast fillet.

Table 3 shows that the color parameters (L*, a*, and b*) of minimally processed chicken breast fillet by wrapping and non-wrapping treatments were not significantly different ($p > 0.05$). Although the results showed no significantly difference, the lightness (L*) of chicken breast fillet by wrapping

with composite SKRC film showed L^* value higher than control (non-wrapping), and the highest value of L^* was wrap plastic. Color changes of chicken breast fillet during storage may be associated with enzymatic and non-enzymatic reactions that cause degradation of myofibrillar proteins [19]. L^* value in meat was associated with protein decomposition in the muscle of the meat [20]. The value a^* shows the redness color of minimally processed chicken breast fillet, while the value of b^* shows the yellowness color. Moreover, the ΔE value provides quantitative information of the overall color difference in each chicken breast fillet by wrapping treatment using wrap plastic or composite SRKC film, i.e. chicken breast fillet with non-wrapping treatment (control). Thus, the difference between control and wrapping application is symbolized by ΔE . The color difference value (ΔE) of chicken breast fillet wrapped by wrap plastic with control color is 3.66 indicating a moderate color difference ($\Delta E = 3-6$). While, the color difference value (ΔE) of chicken breast fillet wrapped by composite SRKC film with control color is 1.50 indicating a small color difference ($\Delta E = 1-3$). In this present study showed that chicken breast fillet wrapped by wrap plastic and composite SRKC film looked fresher when compared to control (non-wrapping). Non-wrapping chicken breast fillets will more easily change color to brown or dark because of direct contact with air. This is caused by the pigment of myoglobin undergoes oxidation to metmyoglobin. While the wrapped chicken breast fillet, either with wrap plastic or composite SRKC film was able to protect or minimize direct contact from outside. This corresponds to the function of the film as a barrier to the mass (such as moisture, oxygen, light, lipids) [21].

4. Conclusion

SRKC-palmitic acid (PA) composite films were successfully prepared and obtained after solvent casting and drying to the formation of an emulsion film. Composite SRKC-based film incorporating 15% w/w of PA demonstrated better water vapor barrier properties as compared to other films with 5% and 10% w/w PA incorporation. Thus, formulation containing 15% w/w PA was used as a wrapping material for film application on minimally processed chicken breast fillet. The application results showed that the incorporation of PA in film caused an effect ($p < 0.05$) on preventing of weight loss significantly compare to control (non-wrapping), however it did not significantly ($p > 0.05$) change the color of minimally processed chicken breast fillet.

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