Physical Quality and Microstructure of Casein-Sodium Tripholyphosphate Edible Film Making

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Abstract. Edible film is a natural packaging that is environmentally friendly (biodegradable) which is strong and elastic, resembling plastic packaging. Cow's milk has component fractionation capabilities that produce a variety of functional ingredients for applications in the food industry. Cow's milk has properties, among others, as an antibacterial agent. Sodium tripholyphosphate (STTP) is one of the additives to improve the integrity of edible films. The purpose of this study was to observe the effect of using different casein on the characteristics of the edible film (film thickness, transparency, water content, solubility in water, and swelling). Edible films were made from different formulations of chitosan, casein, and Sodium Tripholyphosphate (STPP) (1:3:0, 2:3:0, 1:3:3, 2:3:3, 1:10:7, 1:5:1). The manufacture of edible films is carried out through the process of dissolving, mixing, printing, and drying. The results showed that the edible film based on chitosan, casein, and STPP had a significant effect ($P < 0.01$) on water content, water solubility, and swelling, but did not significantly affect the thickness of the edible film and transparency.

Keywords: edible film · casein · moisture content · solubility · swelling

1 Introduction

Packaging is a material used to protect and maintain the quality of a product from damage. The packaging that is often used is synthetic packaging such as plastic. Plastic packaging is a synthetic packaging that is difficult to decompose naturally, causing various environmental problems. Therefore, natural packaging that is similar to plastic is needed, one of which is the use of edible films. Edible film is a packaging made from environmentally friendly natural materials that are strong and elastic. Edible films generally consist of proteins, polysaccharides and lipids [1]. Milk protein is an excellent component used as a basic material for making edible films because it has non-toxic properties [2]. The use of edible films can improve the quality of food ingredients by limiting the transfer of water, CO_2, O_2, lipids, taste and color in food components, improving the quality

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of food ingredients such as antioxidants, antimicrobials, and improving the quality of a product [3].

Bovine milk casein is the predominant protein in cow's milk. Bovine milk has an ingredient fractionation capability that produces different functional ingredients for food industry applications. The total protein in bovine milk is 20% whey protein and 80% casein [4]. Bovine milk has many benefits such as being rich in essential amino acids and containing various proteins with biological activities ranging from antimicrobial to nutrient absorption, acting as growth factors, enzymes, hormones, antibody stimulants and antibodies. Edible film based on milk protein acts as coatings on food or between food components. This edible film also functions for mechanical protection, mass transfer control and sensory properties of a product. The use of bovine milk as a raw material for making edible films has been widely studied. Bovine milk has properties such as antimicrobials. Antimicrobials properties such as nisin, lysozyme, potassium sorbate (K-sorbate), sodium lactate (Na lactate), sorbic acid (SA), e-polylysin (e-PL), natamycin, p-aminobenzoic acid (PABA), lactoperoxidase system (LPOS) and essential oils [5]. There is a difference between making edible film casein with bovine milk. Casein-based edible films were dissolved with aquadest and the addition of a plasticizer using glycerol. This is the same as previous study which reported that edible films were made dissolving casein in 1 g/50 ml of distilled water [6]. Edible film bovine milk is made by dissolving bovine milk with aquadest, Na_2HPO_4 and NaH_2PO_4. This research adds Sodium tripholyphosphate (STTP) to the edible film made. Sodium tripholyphosphate (STTP) is one of the ingredients that can be used as a cross linking agent that can change the starch structure to be stronger and can be used as a reagent that can strengthen the bond between amylose and amylopectin [7]. The purpose of this study was to determine the effect of using casein with the addition of STTP in the manufacture of edible films.

2 Materials and Methods

2.1 Materials

The materials used in the manufacture of edible films include, casein bovine milk (OXOID) and chitosan (Panadia), glycerol (Merck), beeswax (Kembang Joyo), distilled water (Duta Jaya), and 2% acetic acid (CH_3COOH), STPP (Panadia).

2.2 Edible Films Making

The process of making edible coatings is carried out by weighing all solutions, namely bovine milk casein, chitosan and STPP with a ratio of M1 (1:3:0); M2 (2:3:0); M3 (1:3:3); M4 (2:3:3); M5 (1:10:7); M6 (1:5:1). Then first dissolving the casein using distilled water with each ratio, then dissolving the chitosan with distilled water and 2% acetic acid. The casein and chitosan solutions were then heated for 1 h each at a temperature below 50 °C, after 1 h the heating process was carried out and 0.28% glycerol was added to each solution. The casein and chitosan solution were reheated at a temperature below 60 °C for 30 min, after which 0.4% beeswax was added to the solution. The solution was homogenized with a hand mixer for 15 min, then waited until the foam in the solution disappeared. Strained and added STPP solution according to the procedure and homogenized for 30 min.

2.3 Moisture Test

Moisture content was analyzed by oven method which refers to AOAC 2005. The empty cup in the oven at temperature of 105 °C for 24 h then weighed. The sample was weighed (W1) and the sample was put in an oven at 105 °C for 6 h. Cool in desiccator for 30 min. The cup and its contents were weighed again (W2). Calculated by the formula:

$$\text{Moisture content } (\%) = \frac{(W1 - W2)}{(W1 - W0)} \times 100\% \quad (1)$$

2.4 Swelling Degree Test

This is done by cutting the edible film 3 cm × 3 cm. The edible film was dried in an oven at 105 °C and weighed (Wi). Soak the edible film in 50 ml of distilled water at room temperature for 24 h. The edible film was filtered using filter paper and weighed (Wf) [8]. The water absorption capacity is determined using the following equation:

$$\text{Swelling ratio } (\%) = \frac{(Wf - Wi)}{Wi} \times 100\% \quad (2)$$

2.5 Water Solubility Test

Edible film is cut to a size of 2.5 × 2.5 cm. The edible film was dried at 70 °C for 24 h. The sample is weighed and its weight is determined as the initial weight (Wi). Samples were immersed in 50 ml of distilled water for 24 h. The sample was filtered with filter paper and then dried at 70 °C for 24 h. To determine the dry matter that is insoluble in water, the sample is weighed as a final weight (Wf) [9] and [10]. Percent solubility is calculated using the following formula:

$$\%\ \text{Solubility} = \frac{(Wi - Wf)}{(Wi)} \times 100\% \quad (3)$$

2.6 Thickness Test

The edible film was measured using a micrometer at 3 different places, then added together and the average thickness was sought [11].

2.7 Transparency Test

Transparency describes the level of clarity of the resulting film according to the treatment. Transparency testing is done by cutting the film 3 × 3 cm. Place the film on a white sheet of paper so that the transparency of the film can be seen.

2.8 Microstructure Test

The surface and internal structure of edible film were evaluated using scanning electron microscopy. The sample is placed on a double-sided aluminum plate, after which the sample was coated with a metallic powder coating in gold with a plating time of 30 s. Samples were observed at 15 kV voltage and 1000× magnification.

3 Results and Discussion

Table 1. Data on moisture content, degree of swelling, solubility in water, and thickness of the use of casein in the making edible film

Treatment	Moisture Content (%)	Degree of swelling (%)	Solubility in water (%)	Thickness (mm)
M1	33.87 ± 3.97^{ab}	0.72 ± 0.17^{c}	66.47 ± 1.68^{c}	0.07
M2	35.98 ± 1.28^{d}	0.60 ± 0.06^{bc}	75.68 ± 0.49^{f}	0.04
M3	35.04 ± 2.76^{c}	0.55 ± 0.20^{b}	71.09 ± 1.78^{d}	0.08
M4	34.28 ± 2.31^{b}	0.21 ± 0.12^{a}	74.75 ± 3.13^{e}	0.10
M5	33.72 ± 0.67^{ab}	1.24 ± 0.99^{e}	55.10 ± 1.10^{a}	0.14
M6	33.34 ± 0.25^{a}	0.96 ± 0.21^{d}	60.63 ± 2.06^{b}	0.11

3.1 Moisture Content

The results of the study using casein-chitosan edible film with the addition of STPP showed a very significant difference ($P < 0.01$) in the moisture content of the edible film (shown in Table 1). The results were obtained due to the application of various types of casein on edible films. Water content analysis determines the amount of water contained in food ingredients [12]. Edible film with low moisture content will be more resistant to microbiological damage. The high water content will greatly affect the durability of the edible film produced. The addition of lipids such as beeswax content can reduce the water value of edible films because of the hydrophobic nature of lipids [13]. Casein is one type of protein found in milk and has hydrophobic properties, so that the edible films formed has the characteristics of being inelastic, not compact and not transparent [14]. Casein bovine milk consists of 80% casein and 20% consists of whey protein [4].

3.2 Swelling Degree

The results showed that there was a significant difference ($P < 0.05$) on swelling of the edible film (Shown in Table 1). Swelling is the ability of plastic (film) to expand when placed in a solution. The degree of swelling test shows biodegradability and maintains the quality of shelf life of food products [2]. The degree of swelling depends on the materials used to make the edible films [15]. The higher the degree of swelling, the more easily the edible film dissolves in water, even though the absorption is high. On the other hand, the lower the degree of swelling, the easier it is to destroy the edible film in water due to the presence of H_2O that fuses into it [16].

3.3 Solubility in Water

The results of study using casein-chitosan edible film with the addition STPP showed that there was a very significant difference ($P < 0.01$) in the solubility of the edible film in water (can be seen in Table 1). The solubility of edible films is determined by the basic components of the edible film. The solubility of edible films in water can be used as an indicator to measure the resilience, integrity, and ability of edible films in water [6]. Solubility in water is an indication of the hydrophilicity of edible films [17]. The average yield of edible film solubility varies between 60–75%. The highest percentage of edible film solubility was found in the M2 treatment and the lowest edible film solubility percentage was found in the M5 treatment. Most of the casein-based films will dissolve in water which affects the mechanical properties and barrier properties [18]. Edible films with high dissolution causes edible films to be more susceptible to degradation due to decreased binding power. Edible films with low solubility are used as food packaging which generally has high water and Aw content [19]. A high percentage of solubility cannot protect food products from moisture and evaporation [20].

3.4 Thickness

The thickness of the edible film is one of the physical and visual characteristics that can affect permeability and mechanical properties of the edible film. Thickness affects the parameters of transparency, attractiveness and the rate of transmission of steam or gas on the edible film. The results showed that there was no significant difference ($P > 0.05$) in the thickness of the edible film (shown in Table 1). The thickness of the edible film in a formula is influenced by the print media used and the volume of the solution poured into the media. The thickness of the edible film in a formula shows differences or unevenness [21] and [22] stated that the thickness of the edible film can be controlled by reaction parameters such as pH value, settling time and casein concentration. The addition of casein formulations in small amounts can cause the thickness of the edible film forming solution is constant, thus affecting the composition and interactions between components. The thickness of the edible film observed was in the range of 0.14–0.20 mm [11].

3.5 Transparency and Formation

The results of the study describe the level of clarity of the resulting film on transparency. In this study, the results of the transparency test are shown in Fig. 1. Transparency tends to decrease with the addition of plasticizers, meaning that the higher the glycerol content is added, the degree of transparency (clarity) on edible films tends to increase [23]. The

Fig. 1. Transparency and film formation with the use of casein in the making of edible film

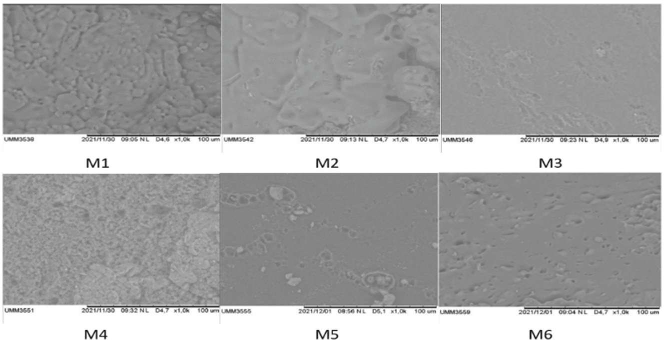

Fig. 2. Appearance of the edible film microstructure with the use of casein in the making of edible film

results of the analysis of edible film transparency based on the M1 to M5 treatments were not significantly different ($P > 0.05$). Transparency values show similar results to transmittance, so films that are less transparent can be used as food shields against light and prevent lipid oxidation [24]. Packaging transparency is usually more attractive to consumers because it is more visually informed about food or products before buying and food ingredients can look appetizing [25].

3.6 Microstructure

The results of the SEM test showed that the appearance of the microstructure of the edible film varied from slightly flat to very uneven with varying number and size of the cavities/pores varying (See Fig. 2). Microstructures produced using modified casein exhibiting varied appearances and morphology [6]. The composition of the film affects the compactness of the microstructure and may occur due to a strong cohesive force that emerges during slow material [26].

4 Conclusions

It can be concluded that edible films made from casein and chitosan with the addition of STPP have a significant effect on water content, degree of swelling and solubility in water, but have on significant effect on the thickness of the edible film and the transparency. In the research results, it was found that the degree of swelling had the highest value in the M5 treatment and lowest average was found in the M4 treatment. The highest percentage of edible film solubility was found in the M2 treatment and the lowest percentage of edible film solubility was found in the M5 treatment. The highest edible film thickness was in the M5 treatment and the lowest thickness was in the M2 treatment.

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