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Development of a Functional Edible Film Based on Avocado Seed Starch Reinforced with CMC and Clove Extract for Traditional Dodol Packaging

Linda Maulida[1] Muhammad Iqbal Endy Wicaksana[2], Yulia Nurul Ma'rifah[1], Nova Sari Bulkis[1], Debby Aulia Nur Islami[1], Iryanti Fatyasari Nata[1,2,3,] ✉

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[1] Department of Chemical Engineering, Lambung Mangkurat University, Banjarbaru 70714, Indonesia
[2] Department of Environmental Engineering, Lambung Mangkurat University, Banjarbaru 70714, Indonesia
[3] Integrated Laboratory, Lambung Mangkurat University, Banjarbaru 70714, Indonesia

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Abstract

Edible film is a thin layer, which uses natural materials that can be consumed and used as eco-friendly food packaging. Avocado seed starch is used as the principal matrix of edible film and carboxymethyl cellulose (CMC) is applied as a filler, and clove extract is included as an antibacterial. This study aimed to measure the role of CMC and clove extract as antibacterials on edible film in terms of thickness, tensile strength, elongation at break, and its use as a packaging medium in South Kalimantan dodol snacks in general. Moreover, to define the edible film in terms of morphological structure, functional groups, water vapor permeation, degradation, and antibacterial properties. This study the concentration of CMC (30, 35, and 40% w/w) and clove extract (5, 10, and 15% v/v) were selected as experimental variables, and the mixture were prepared by adding 100 mL of distilled water and 30% glycerol (w/v) under constants stirring. The avocado seed starch (4 g) was then added, and heated at 80 °C during 1 h; it was moulded and baked at 55 °C during 12 h. This research anticipates that edible film will hold promise as a biodegradable packaging that works to preserve food as well as lengthen the shelf-life. The optimal concentration and thickness of 0.136 mm, tensile strength of 4.947 N/mm^2, elongation at break of 92.3%, water vapor transmission of 0.212 g/m^{2}h were the best results of the edible film test. The resulting edible film is usable as an environmental friendly food as wrapping material.

INTRODUCTION

With the increasing population, food demand is also increasing, and waste is also increasing. The use of non-biodegradable or synthetic plastic packaging is dominant in the Indonesian food industry due to its flexibility, diverse applications, and relatively low cost (Putri et al., 2019). The food and beverage industry is a major contributor to plastic waste (Musdar, 2020). One solution to reducing environmental impact is the use of biodegradable plastic. This product has been developed as a more environmentally friendly and safe packaging alternative for food products. One commonly used form of environmentally friendly or biodegradable packaging is edible film.

Edible film is a thin film whose thickness is less than 0.25 mm, and can be consumed directly. Its major role is gas exchange control, moisture retention, and preservation of valuable ingredients in food items (Putri et al., 2019). Edible film application in the food sector is a strategic move to advance the concept of sustainability alongside fulfilling the consumer needs to have and buy safe and eco-friendly products (Marlina et al., 2021). The choice of raw materials is a key aspect in the production of edible films. A possible and not very common ingredient is avocado seeds (Musdar, 2020). Avocado seed starch has 43.31% amylose,

✉ Corresponding author:
E-mail: ifnata@ulm.ac.id

and this is greater than corn starch that has 27% (Masato and Wahyuningtyas, 2022). The biodegradable characteristics of the film are influenced by the role of fillers, which have a positive effect on the edible film's characteristics. Furthermore, clove essential oil contains a high content of eugenol (76.8%), β-caryophyllene (17.4%), α-ihumulene (2.1%), and eugenyl arihetate (1.2%), as the main antimicrobial pathogenic compounds (Fahrullah, 2021).

Previous research by Putri et al. (2019) showed that increasing the concentration of 5-20% increased film thickness about 0.098-0.142 mm. The water uptake also increased with increasing CMC concentration, indicating that better water resistance. Water solubility decreased with increasing CMC, with tensile strength peaking at 10% CMC at 3.56 N/mm^2, and elongation increasing to 18.3% at 15% CMC. Nevertheless, the resulting edible film met the Japanese Industrial Standard (JIS) for tensile strength, although elongation did not reach the 70% required for food packaging. Clove extract can have beneficial effects on microbiological properties and clove extract at a concentration of 5 percent can be used as an antibacterial agent to prevent the growth of different microorganisms such as bacteria (Staphylococcus aureus and Escherichia coli), fungi, and yeast (Fahrullah, 2021). Research has been conducted on the manufacture of edible film from jackfruit seed starch with sorbitol and CMC. The edible film results in cracks and incompletely dissolved starch particles on the film surface (Indriani et al., 2023). Furthermore, other research indicates that the characteristics of edible film containing glycerol can cause it to break easily due to its low tensile strength (Ermawati and Haryanto, 2020). Therefore, a combination of edible film components is needed to improve the quality and functionality of food packaging.

This research assessed the incorporation of CMC and clove extract in the quality of edible film, such as thickness, tensile strength, elongation at break, characterization, functional group analysis, and percentage of water absorbed test for future usage as a packaging medium to package dodol (a snack product). This study should help in producing packaging materials that are eco-friendly and natural to reduce the use of single-use plastics worldwide.

MATERIALS AND METHOD

Materials

The materials used included avocado seeds obtained from juice trader waste in Banjarbaru, Kalimantan Selatan, Carboxy Methyl Cellulose (CMC), glycerol, and cloves.

Avocado Seed Starch Preparation

The Indriani et al. (2023) obtained starch of the avocado seeds by peeling and thoroughly washing the avocado seeds. Seeds were cut 1-5 mm thick and mixed with the avocado seeds in a ratio of 1:3 with a distilled water. The slurry was filtered to achieve the separation between the starch and the pulp. Distilled water was then added to the pulp in the ratio of 1:3. The filtrate was left to settle overnight and the starch precipitate dried in the oven at 60 °C. Dried starch was sieved and ground to go through a 60-mesh sieve.

Clove Extract Preparation

Clove powder, 5 g (± 60 mesh) was put in 100 mL of distilled water. The solution was warmed under 400 W in the microwave at 80 °C within 5 minutes. The mixture was then heated and then cooled and filtered using filter paper and left to reach room temperature in order to extract the clove extract.

Edible Film Preparation

CMC (30, 35, and 40% w/w) was put in the presence of 100 mL of distilled water and 30% w/v glycerol. The solution was stirred with the magnetic stirrer after 10 minutes at 60 °C. After homogenizing it, 4 g of avocado seed starch was put in and heated at 80 °C stirring at 300 rpm. The solution was poured in the molds after it had attained room temperature and dried in an oven at 55 °C and was allowed to dry for 12 h. Thickness, tensile strength and elongation of the formed edible film were then tested in each of the variables. The physical test had the best CMC composition, which was chosen as the CMC concentration in the following test. The identical procedure as the one above but with a constant CMC concentration was added with clove extract with modification of 5, 10, and 15% v/v. The follow up experiment of the edible film product was again tested in the areas of thickness, tensile strength, elongation at break. The most promising outcomes of the variant of the clove extract were subject to morphological characterization and functional group analysis, and water absorbed test that recommendation for food packing.

Analysis and Characterization

The transmission of the water vapor was determined by measuring the edible film into the 2x2 cm pieces and using them in the 1st container that contained the distilled water and the container 2 that contained the silica gel. The samples were dried in the oven at 105 °C over a period of 5 h, desiccated over 24 h, and finally weighed. The equation to estimate the transmission of water vapor can be taken as:

$$\mathrm{WVP} = \frac{\Delta \mathrm{W}}{\mathrm{t \, x \, A}} \quad (1)$$

Where, W is the change in edible film weight after 24 h, t is the measurement time (24 h) and A is the film surface area (m^2).

The tensile strength of the edible film was tested using a Torsee's Electronic System Universal Testing Machine at a speed of 20 mm/min and a load capacity of 100 kgf. Each sample was tested twice, and the average value was taken. Elongation at break was calculated as the difference between the maximum length of the film from the moment of tensile force until it broke and its initial length. Film thickness was measured with a digital micrometer with an accuracy of 0.01 mm at 5 different points.

The sample surface structure was observed with a Scanning Electron Microscope (SEM) Carl Zeiss Evo 10 to determine the surface morphology, and functional groups was using an Fourier Transform Infra Red (FT-IR) spectrophotometer (Eco-ATR Alpha II Bruker). Its optical system uses laser radiation interfered with infrared radiation to provide a complete and more high-quality signal for functional group identification.

RESULT AND DISCUSSION

The Effect of CMC Concentration on Edible Film

The initial control experiment with avocado seed starch in the absence of CMC and clove extract failed to form a continuous and manipulable film since the matrix was brittle and fractured hence this formulation could not be further characterized. Thickness of film is influenced by the CMC added to edible film products prepared using avocado seed starch. Fig. 1 (inset) indicates edible films formed of avocado seed starch containing 30, 35 and 40% w/w CMC. All of three CMC variations produced brownish edible films. The higher the CMC concentration, the deeper the brown color, which originates from the starch. Edible film thickness is an important characteristic in determining the suitability of edible film for food packaging, as it affects its physical and mechanical properties. Edible film thickness at various CMC concentrations is shown in Figure 1.

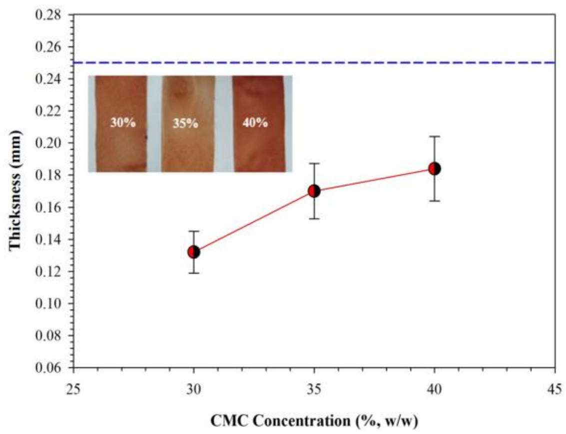

Figure 1. Edible Film Thickness in Variations in CMC Concentrations of 30%, 35%, and 40% (w/w). Operating Conditions: Total Volume 100 mL, 30% Glycerol, and Temperature 80 °C.

According to Figure 1, the thickness measurements of the edible film with variations in CMC concentrations of 30%, 35%, and 40% w/w were 0.132 mm, 0.172 mm, and 0.184 mm, respectively. The highest thickness was found for the 40% CMC edible film at 0.184 mm, while the 30% CMC had the lowest thickness at 0.132 mm. Edible film thickness increased with increasing CMC concentration, this is because CMC is able to form complex solutions to increase viscosity (Putri et al., 2019). The thickness test results, in accordance with the Japanese Industrial Standard (JIS), stipulate that the maximum standard thickness of edible film is 0.25 mm.

Tensile strength and elongation at break are critical mechanical characteristics for determining the quality and performance of edible film. Based on Figure 2, the tensile strength test results for edible film with variations in CMC content of 30%, 35%, and 40% (w/w) were 2.493

N/mm^2, 4.047 N/mm^2, and 6.783 N/mm^2, respectively.

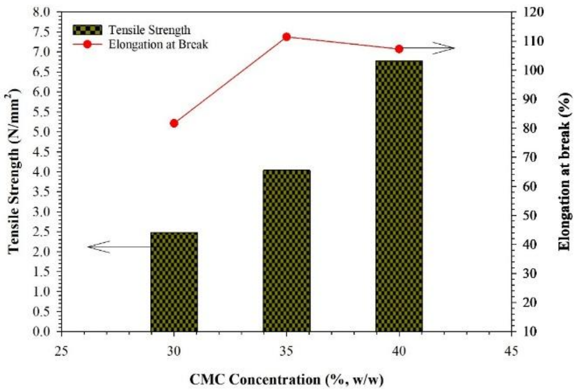

Figure 2. Tensile Strength and Elongation at Break of Edible Film with Variations in CMC Concentrations of 30%, 35%, and 40% (w/w). Operating Conditions: Total Volume 100 mL, 30% Glycerol, and Temperature 80 °C.

The highest tensile strength value was 6.783 N/mm^2 at a 40% CMC concentration, and the lowest value was 2.493 N/mm^2 at a 30% CMC concentration. The tensile strength of edible film tends to increase with increasing CMC concentration. These results align with research by Indriani et al. (2023), which found that tensile strength increases with increasing CMC content. Increasing CMC concentration results in a thicker bioplastic matrix due to the increased amount of polymer constituents. As a result, the force required to break the specimen increases, resulting in an increase in tensile strength (Ningsih et al., 2019). The tensile strength test results are in accordance with the JIS standard minimum tensile strength value of edible film of 3.92 N/mm^2. The elongation at break test is a measurement of the percentage increase in the length of a biodegradable film from its initial length to the point where it breaks due to pulling (Elean et al., 2018). Based on Figure 2, the results of the elongation at break test on edible films with CMC variations of 30%, 35%, and 40% (w/w) obtained values of 81.68%; 111.43%; and 107.24%, respectively. The elongation at break value will increase along with the increase in CMC (Nurindra et al., 2015). The elongation at break test results shows an increase from 30% to 35% CMC concentration, but at a concentration of 40% there is a slight decrease. This is because the polymer matrix becomes denser so that flexibility is reduced (Putri et al., 2019). The elongation at break test results also met JIS standards, with a minimum elongation at break value of 10% for edible film. Based on the tensile strength and elongation at break tests, edible film with 40% CMC was determined to be the best composition.

Morphological observations using SEM with varying CMC concentrations are expected to improve the physical quality of the edible film. Figure 3 shows clumps with each increase in concentration, and the volume of the clumps increases with each increase in CMC concentration.

These clumps represent bonds between the CMC matrix and starch, indicating that the greater the bond, the more elastic the edible film. The evenness or unevenness of the surface depends on the mixing process; to produce an edible film with a good surface, the matrix and plasticizer must be well mixed.

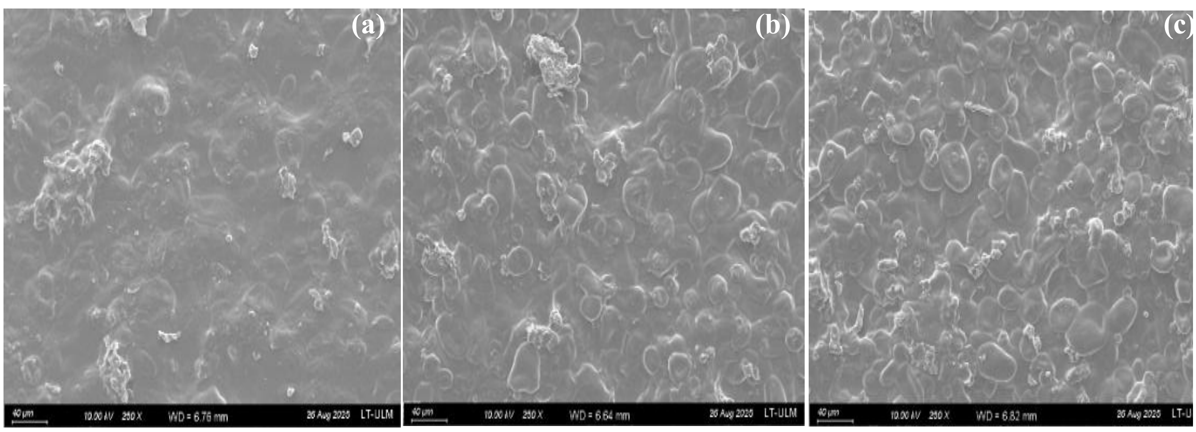

Figure 3. SEM Images of Edible Film with Addition of CMC (a) 30%, (b) 35%, and (c) 40%. Operating Conditions: Total Volume 100 mL, 30% Glycerol and Temperature 80 °C.

Effect of Clove Extract Addition on Edible Film Products

The addition of clove extract affected the characteristics of avocado seed starch edible film. The clove extract concentrations used varied at 5%, 10%, and 15% v/v, with the resulting CMC composition of 40% shown in Figure 4 (inset). The resulting edible film was brown due to the natural color of the starch and clove extract. Cloves contain eugenol, a compound that can inhibit the growth of pathogenic bacteria that cause food spoilage, such as Bacillus cereus, Escherichia coli, and Staphylococcus (Sulaiman et al., 2023). The amount of clove extract added affects the characterization of the edible film. When clove extract is added, the edible film becomes darker.

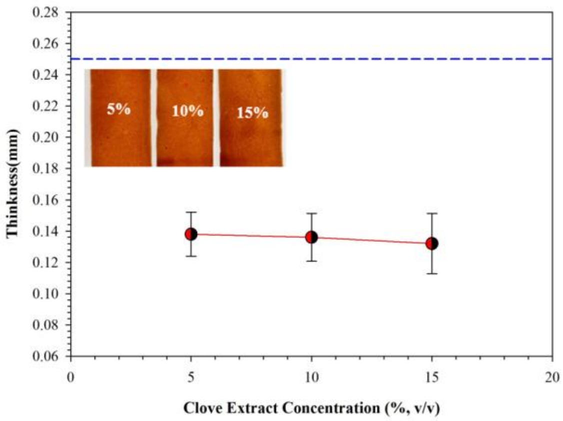

Figure 4. Edible Film Thickness in Variation of Clove Extract Concentrations of 5%, 10%, and 15% (v/v). Operating Conditions: Total Volume 100 mL, 40% CMC, 30% Glycerol, and Temperature 80 °C.

Based on Figure 4, the thickness of the edible film with clove extract concentrations of 5%, 10%, and 15% v/v is 0.138 mm; 0.136 mm; and 0.132 mm, respectively. The more clove extract added, the thinner the edible film. This is because the more clove extract added, the lower the water content of the edible film. Clove extract can limit the interaction of polysaccharides with water by hydrogen bonding, resulting in a decrease in the water content of the edible film (Nissa and Myra, 2021). All thickness values have met JIS requirements.

The tensile strength test on the edible film with clove extract added in Figure 5 at concentrations of 5%, 10%, and 15% v/v was 6.425 N/mm^2; 4.977 N/mm^2; and 3.103 N/mm^2, respectively. The tensile strength decreased with increasing clove extract concentration, as the hydrophobic nature causes the edible film to stiff (Nissa and Myra, 2021). The tensile strength test results comply with the JIS standard minimum value of 3.92 N/mm^2. The elongation at break test of the edible film with the addition of clove extract in Figure 5 showed 91.22%, 92.30%, and 50.18% (v/v) of clove extract, respectively. The elongation at break test results showed an increase with the clove extract concentration, but decreased at the 15% concentration. This is due to the excessive hydrophobicity of the clove extract, which makes the film matrix stiffer. This disrupts the bonds between starch molecules and reduces flexibility (Nissa and Myra, 2021). Based on the tensile strength and elongation at break tests, the edible film with 10% clove extract was determined to be the best.

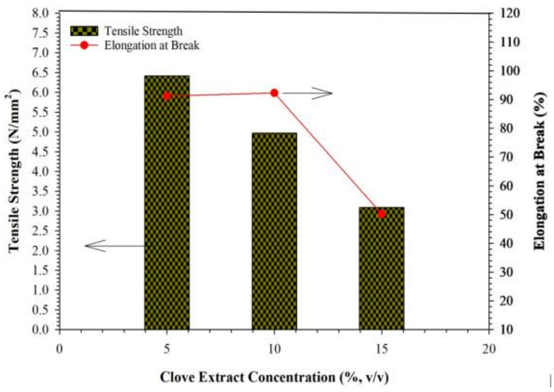

Figure 5. Tensile Strength and Elongation at Break of Edible Film with Clove Extract Concentrations of 5%, 10%, and 15% (v/v). Operating Conditions: Total Volume 100 mL, 40% CMC, 30% Glycerol, and Temperature 80 °C.

Morphological observations using SEM for clove extract concentrations of 5%, 10%, and 15% v/v are shown in Figure 6. Increasing the volume of clove extract did not result in significant changes in the morphological structure, the addition of clove extract only affected the physical properties. While the uneven morphological structure depends on the gelatinization process, in addition the homogeneity of the matrix and plasticizer affects the morphological structure of the edible film.

The FT-IR spectrum of the edible film with CMC and clove extract is shown in Figure 7. Based on IR spectra, the clove extract shows a functional group at 3269 cm^{-1} as a hydroxyl group, indicating an intermolecular H bond. This functional group

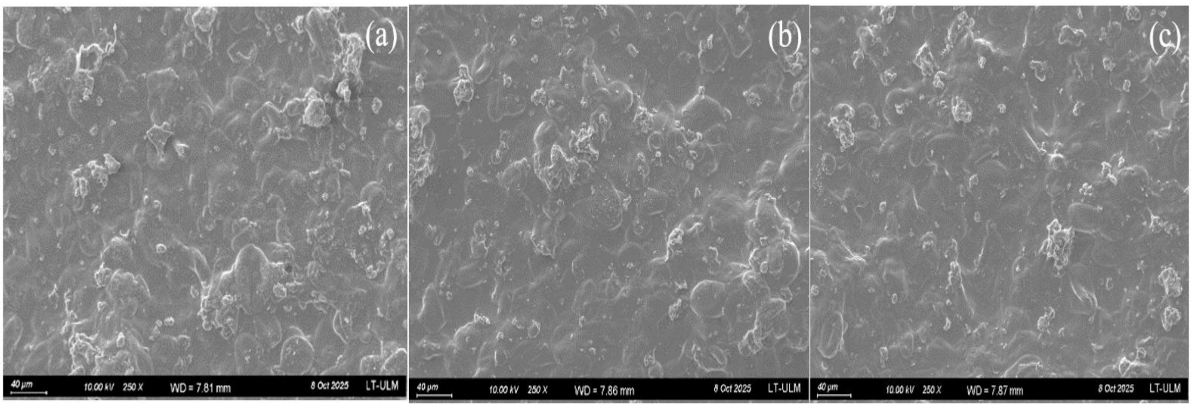

Figure 6. SEM Images of Edible Film with Clove Extract Addition (a) 5%, (b) 10%, and (c) 15% (v/v). Operating Conditions: Total Volume 100 mL, 40% CMC, 30% Glycerol, and Temperature 80 °C.

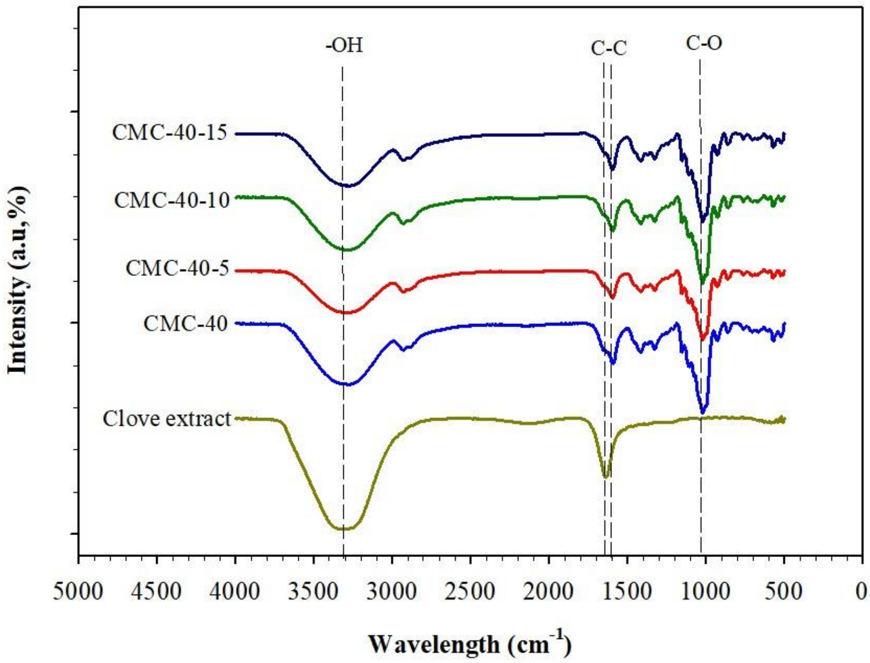

Figure 7. FT-IR of Edible Film with Variations in Clove Extract Concentrations of 5%, 10%, and 15% (v/v). Operating Conditions: Total Volume 100 mL, 40% CMC, 30% Glycerol, and Temperature 80 °C.

has the highest absorption for clove extract. The absorption at 1590 cm^{-1} indicates the presence of a C-C bond as the framework of the molecular structure. The absorption at 1020 cm^{-1} indicates the presence of a C-O stretching for alcohol/ether.

The water vapor transmission (WVT) values of the edible film in Figure 8 for 5%, 10%, and 15% v/v clove extract are 0.277 g/m^2h; 0.212 g/m^2h; and 0.032 g/m^2h, respectively. These results indicate that the water vapor transmission rate decreases with increasing clove extract concentration. This is in line with the opinion of Nissa and Myra (2021), who stated that clove extract is a phenolic compound containing an alcohol group in its chemical structure, making it a good barrier because the hydroxyl group has a lower affinity for water. Water vapor transmission should be considered as a measure of a material's ability to pass water vapor. Based on research, the best concentration of edible film was 10% clove extract, considering tensile strength and elongation at break. When clove extract was added at lower contents (5-10%), there was no negative effect on the elongation at break (EAB) values were relatively high (~90%). This indicates that the phenolic constituents of clove extract at these concentrations are well dispersed in the matrix and can have an analogous effect to secondary plasticizers to increase intermolecular spacing and keep chains mobile. The same has been found in polysaccharide-based films with low concentrations of plant extracts, where there is limited interaction between hydrogen bonding, which enhances cohesion of the matrix but does not inhibit polymer mobility too much (Venkatachalam et al., 2023). However, when in a more concentrated solution (15%), a larger effect on EAB (~50%) was observed, and it also shows the change to a more rigid and brittle film structure. This is attributed to the fact that there are more intermolecular interactions between phenolic compounds and hydroxyl groups of starch and CMC. The forces lead to finer and more tightly packed network formation when the concentration is high and effectively decrease the free volume and restrict the movement of the polymer chains. In some cases, phenolic compounds also may result in crosslink-type interactions that will stiffen the matrix, reducing its extensibility. The observation is consistent with results of previous studies that excessive use of bioactive extracts or essential oils leads to rigidity of films and reduced elongation due to reduced chain dynamics (Kanmani and Rhim, 2014).

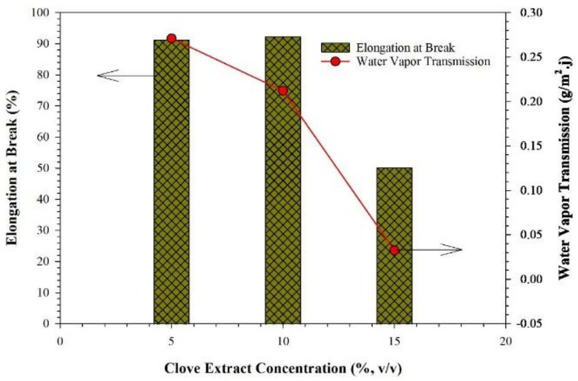

Figure 8. Water Vapor Transmission Values of Edible Film with Variations in Clove Extract Concentrations of 5%, 10%, and 15% (v/v). Operating Conditions: Total Volume 100 mL, 40% CMC, 30% Glycerol, and Temperature 80 °C.

WVT was also shown to exhibit the same concentration effect, decreasing significantly at 15% clove extract. This improvement of barrier properties can be attributed to two factors. First of all, the hydrophobicity of constituents of the clove extracts, particularly eugenol, reduces the hydrophilicity of the film matrix, hence reducing the water adsorption and diffusion. Second, the tight interaction between molecules causes the creation of a smaller polymer network, thus decreasing the amount of free space to move water vapor. This causes the diffusion pathway to become more tortuous, resulting in reduced permeability. Similar studies demonstrated that the addition of phenolic-rich extracts in starch-based films enhances the moisture barrier property, owing to enhanced compactness of the matrix and hydrophobic interactions (Atarés & Chiralt, 2016, Pranoto et al., 2005).

The combination of avocado seeds, CMC, and clove extract produces an edible film with effective barrier properties against external factors. Food shelf life was observed by applying an edible film coating to dodol left exposed to air and its durability in the refrigerator. Observations of dodol can be seen in Figure 9.

The Observations revealed that the dodol only deteriorated on the 14th day, when mold began to appear on the dodol. The sample with the highest mold growth was the one with a 15% clove extract concentration. Despite the presence of more mold, the dodol remained in good condition and odorless. This indicates that clove extract at this concentration is effective in inhibiting the growth of spoilage bacteria that cause rancidity, but is less effective against mold. Dodol stored in the open air is exposed to higher and fluctuating room temperatures, which can accelerate microbial growth and other degradation reactions. Even when coated with edible film, warmer temperatures and

Figure 9. Dodol coated with edible film and left in the open air.

Table 1. Characterization of edible film from avocado seed starch, CMC and clove extract.

Characteristic	Edible Film from experiments	Japanese Industrial Standard (JIS) 1975
Tensile strength, N/mm^2	4.947	min 3.92
Elongation at Break, (%)	92.30	min 10-50
Thickness, mm	0.136	max 0.25
Water vapor transmission, g/m^2h	0.212	max 10

higher oxygen exposure in the open air can accelerate product deterioration. Observations were also made on dodol coated with edible film and stored in a refrigerator at ±4 °C, and the results showed that the dodol remained in good condition and did not deteriorate. Observations were made for 14 days, and the dodol remained in good condition. Storage temperature plays a crucial role in determining the shelf life of food products. Storage at low temperatures can slow the rate of chemical reactions and microbial activity that cause spoilage.

The results of the mechanical and barrier properties clearly demonstrated a trend with increasing concentration of clove extract, with the formulation containing 10% of clove extract showing the most balanced performance in terms of elongation at break (EAB) and water vapor transmission (WVT). At this concentration, the film showed good flexibility (EAB) with significant reduction in WVT, indicating an optimum compromise between mechanical integrity and moisture barrier properties. However, these physicochemical findings were only correlated with qualitative shelf-life observations using dodol as a model food and had not been supported by quantitative analyses such as microbial count, moisture content, pH measurement or sensory evaluation. These analytical parameters will be required in future studies to develop a more complete relationship between the film properties and real food preservation performance.

The analysis results of edible films made from avocado seed starch with 40% CMC and 10% clove extracts compared to JIS are shown in Table 1.

CONCLUSION

Based on the research results the best concentration of Carboxy Methyl Cellulose (CMC) addition in the manufacture of edible films is at a concentration of 40% and 10% clove extract with a thickness of 0.136 mm, tensile strength of 4.947 N/mm^2, elongation at break of 92.3%, water vapor transmission of 0.212 g/m^2h. The resulting edible film can be applied as an environmentally friendly coating material for dodol snacks.

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