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Effects of Glycerol Plasticizer on Physical Characteristic of Whey-Konjac Films Enriched with Clove Essential Oil

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HIGHLIGHTS

- Tensile strength with addition of 0, 5, 10, and 15% clove essential oil were 9.16, 7.9, 7.1, and 6.52 N, respectively.
- Higher level of clove essential oil can increase the elongation and WVTR of the whey-konjac film.
- The concentration of 5% clove essential oil resulted in the best physical properties of film.

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Acronyms and abbreviations
SEM=Scanning Electron Microscope
WVTR=Water Vapor Transmission Rate

ABSTRACT

Background: Edible film is a food packaging that can be eaten directly and have protection function of outside contamination. The purpose of this research is to know how about the effect of using a glycerol plasticizer to film whey-konjac edible films that enriched with clove essential oil.

Methods: A completely randomized design was used by different clove essential oil including P_{10}, 0% clove essential oil; $P_{10}C_1$, 5% clove essential oil; $P_{10}C_2$, 10% clove essential oil; and $P_{10}C_3$, 15% clove essential oil. The present study investigated the physical characteristics, including tensile strength, elongation, Water Vapour Transmission Rate (WVTR), and microstructur film. Data were statistically evaluated using an Analysis of Variance (ANOVA) in Statistical Program for Social Science (SPSS) 16.0.

Results: Results showed that the addition of glycerol plasticizer enriched with clove essential oil had no significant difference (p>0.05) to value of tensile strength, elongation, and WVTR of whey- konjac edible film. The value of tensile strength with the addition of clove essential oil at doses of 0, 5, 10, and 15% were 9.16, 7.9, 7.1, and 6.52 N, respectively. The concentration of 5% clove essential oil resulted in the best physical properties of film with a tensile strength 7.90 N, elongation 64.0%, and WVTR 8.12 g/mm^2/day.

Conclusion: Use of clove essential oil with different concentrations had no effect on tensile strength, elongation, and WVTR; but the addition of clove essential oil had promising potential to improve the physical characteristic of whey-konjac edible films.

Introduction

Synthetic plastic polymer is a non-biodegradable polymer that has been generally used in food packaging that has a relatively low cost (Kamsiati et al., 2017). However, there are concerns that the waste generated cannot be decomposed in nature. Food packaging from synthetic plastics causes environmental pollution,

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producing the waste products, and very difficult to degrade naturally (Siskawardani et al., 2020). The development of food packaging technology is currently moving towards better products, increasing of the consumer demand for packaging products that are natural, and biodegradable rather than synthetic packaging materials (Arifin et al., 2022), with increasing of environmental and food safety issue for development of the research in biodegradable packaging (Dick et al., 2015; Fahrullah, 2021; Fahrullah and Ervandi, 2021; Maniglia et al., 2015; Maruddin et al., 2018; Otoni et al., 2017; Spotti et al., 2016; Sukhija et al., 2016).

Edible film is a food packaging technology which defined as a thin polymer layer that provides protection against gas and moisture contamination by acting as a protein barrier, while it can also be consumed together with the packaged food product (Hammam, 2019). Because of its safety and range of sources, edible film packages with natural polymer materials that can be used for biomedical and medicinal purposes (Daza et al., 2018; Mohammadi Nafchi et al., 2017). However, the functional characteristic of materials that are commonly used as synthetic materials can lead to several drawbacks including availability and cost issues (Liang and Wang, 2018). Therefore, it needs a research to explore biological sources for food product packaging applications. Various biopolymer compounds in the manufacture of edible films include lipids, proteins, polysaccharides, and their derivatives which can be obtained from animals, plants, and microorganisms (Zhang et al., 2016).

The use of protein to produce edible films can be obtained by using whey protein. Whey is known as a by-product of milk processing that has high nutritional and functional value (Jiang et al., 2019, 2020). It has a potential as a basic ingredient for manufacture of edible films because it has superiority in creating films such as transparent, can block gases, aromas, and oils, odorless, elastic, and can maintain the aroma of the packaged product (Maruddin et al., 2018; Umaraw and Verma, 2017). The increasing of physical characteristic of whey-based edible films can be possible with using other polymers such as hydrocolloids (proteins and polysaccharides), fats, or their combination (Fahrullah et al., 2020a). One of the polysaccharides that actions together with whey protein in producing edible films is konjac, where it contains a glucomannan component which is a soluble dietary fiber (Fahrullah et al., 2020b). The physical characteristic like gelation in edible film is expected to form with combination of whey and konjac (Fahrullah et al., 2020a). Composite edible films give better function than edible films made from only one particular polymer (Serna and Filho, 2015).

Problems that often occur in edible films are brittleness, easy to break, and hard texture (Maruddin et al., 2018). To produce efficient edible films, the characteristics of edible films must be optimized for commercial applications by means of the plasticization process. In making of edible film need a plasticizer for reducing protein chain interactions, disrupting hydrogen bonds to increase film flexibility, and Water Vapour Transmission Rate (WVTR) (Farhan and Hani, 2017). To prevent the water loss, it is possible to add glycerol to polymer bonds and create spaces between polymer chains (Fahrullah et al., 2020a). Research by Vieira et al. (2011) showed that the addition of a plasticizer prevents the brittleness of the film during handling and storage. The physical characteristics of edible films with the addition of clove oil depend on compatibility, preparation techniques, and drying processes (Galus and Kadzińska, 2016). So, flotation of clove essential oil in the film improves its quality (Silva et al., 2020). In this research, there is difference from others in the drying process only uses room temperature and does not use an oven. So, the aim of this research was to produce whey-konjac edible films that enriched with clove oil and to determine effect of using a glycerol plasticizer on physical characteristics of them.

Materials and methods

Materials and instruments

The materials are whey powder (CV. Makmur Sejahtera, Indonesia), konjac (Prima Food, Indonesia), glycerol plasticizer (CV. Makmur Sejahtera, Indonesia), clove oil (CV. Makmur Sejahtera, Indonesia), distilled water, aluminum foil, silica gel, plastic wrapping, label paper, tissue, and clean water. The main instruments used included erlenmeyer (Pyrex, Indonesia), thermometer (Gelsonlab HSGT-067, China), magnetic stirrer, hot plate stirrer (FAITHFUL SH-2, China), desiccator, digital gauge HF 500, and Scanning Electron Microscope (SEM; JEOL JSM 5,310 LV Model, Japan).

Sample preparation

Samples were collected in January 2019. The first step was preparation stage with making a whey-konjac edible film solution. Eight percent (w/v) whey powder was mixed with 0.5 g (w/v) konjac, and then distilled water was added until the solution reached a final volume of 25 ml. The whey-konjac film solution was added with 35% glycerol plasticizer, and then the solution was heated at a temperature of 90±2 °C on a hot plate and stirred using a magnetic stirrer at 250 rpm for 30 min. It was poured into a petri dish and then placed at room temperature for 24 h. The finished edible film was packaged using aluminum foil for two days before testing (Modification from Fahrullah et al., 2020b; Maruddin et al., 2018).

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Tensile strength

The sample of edible film was cut with the size of 8×3 cm, then it hooked to horizontally to the clamp with diameter of 1.5 cm in each long side, and the maximum tensile strength was measured when the film showed signs of damage during the pulling process (Wittaya, 2013).

Elongation

The sample of edible film was cut with the size of 10×5 cm, then it determined using a universal instrument tensile strength meter and stretched at a speed of 50 mm/min (ASTM D882-18, 2018), and then stretched at a speed of 50 mm/min. The formula used for film elongation was adopted from Wardana and Widyaningsih (2017):

$$\text{Elongation (\%)}=\frac{L}{L0}\times 100\%$$

L is the film length at break (mm) and L0 is the initial film length (mm).

WVTR

The WVTR was measured by cutting a circular film with a diameter of 2.8 cm. The film pieces were stored in a glass containing 3 g of silica gel and put in a desiccator, and then measurements were taken every 24 h for 5 days. The WVTR is expressed in units of g/mm^2/day using the formula (ASTM E 96-95, 1995):

$$\text{WVTR}=\frac{n}{t\times A}$$

n is the change of weight (g), t is time (days), and A is the surface area of the edible film (mm^2).

Microstructure of the film

The microstructures of the edible film were tested using an electron microscope of SEM JEOL JSM 5,310 LV. The edible film was prepared with the size of 0.5×0.5 cm, and then it coated with carbon and gold. The ready sample was then placed on the SEM device for microstructural observations.

Statistics analysis

Data processed using completely randomized design with three treatments and five replications. The treatment used by different clove essential oil included P_{10}, 0% clove essential oil; $P_{10}C_1$, 5% clove essential oil; $P_{10}C_2$, 10% clove essential oil; and $P_{10}C_3$, 15% clove essential oil. Data were statistically evaluated using an Analysis of Variance (ANOVA) in Statistical Program for Social Science (SPSS) 16.0. The significant difference between means was verified using the Duncan test (*p* value <0.05).

Results

Tensile strength

The use of clove essential oil had no a significant difference (p>0.05) in the film tensile strengths. The value of tensile strength with the addition of clove essential oil at doses of 0, 5, 10, and 15% were 9.16, 7.9, 7.1, and 6.52 N, respectively (Figure 1).

Elongation

The use of clove essential oil had no a significant difference (p>0.05) in the film elongations. Percentage value of the edible film elongation showed that the higher addition of clove essential oil was higher the elongation of the film, but had time decreasing in addition of 10% clove essential oil and increasing again at a concentration of 15% (Figure 2).

WVTR

The use of clove essential oil had no a significant difference (p>0.05) in the film WVTR's. However, it happens the increasing to WVTR value with addition of clove essential oil compared the controls (Figure 3).

Microstructure film

Observation of this microstructure was an important element to determine the characteristic of the film. The visible appearance showed that whey-konjac film was not torn, looked transparent, was in good condition, and was in an orderly and neat shape. The higher addition of clove essential oil was more opaque the color of resulting film. The use of 0% clove essential oil produced a structure with a flat surface; the mixture of whey solution with konjac was evenly distributed. Microstructure of edible film whey-konjac with addition of clove essential oil produced a less homogeneous surface structure.

Discussion

The values of tensile strength showed that the higher use of clove oil was lowers the tensile strength value. The use of clove essential oil had no a significant difference (p>0.05) in the film tensile strengths. It was very important to noticed because it often subjected to external stresses and must maintain its structural integrity during the packaging, transportation, and storage processes (Xu et al., 2020). The tensile strength value in this research showed that the more addition of clove essential oil to the film mixture, then it was produced the lower value. During the film making process, whey and konjac polymers interact each other through hydrogen bonds, both of which are responsible for the fabrication of the network

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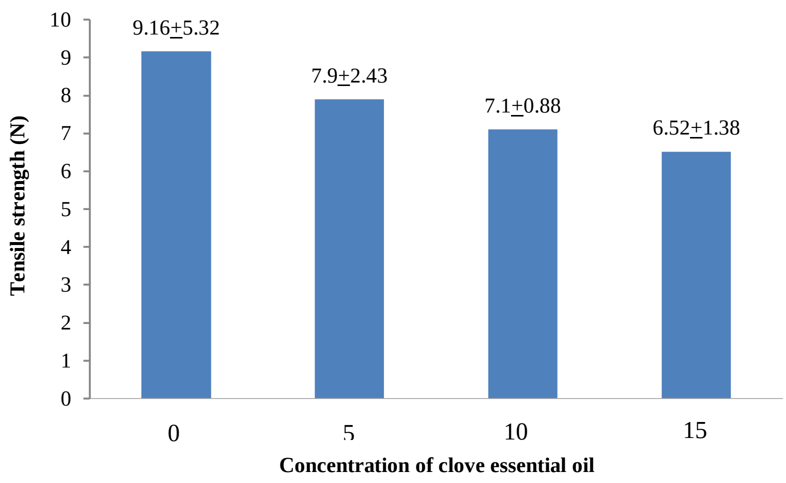

Figure 1: Tensile strength of whey-konjac film with the addition of clove essential oil

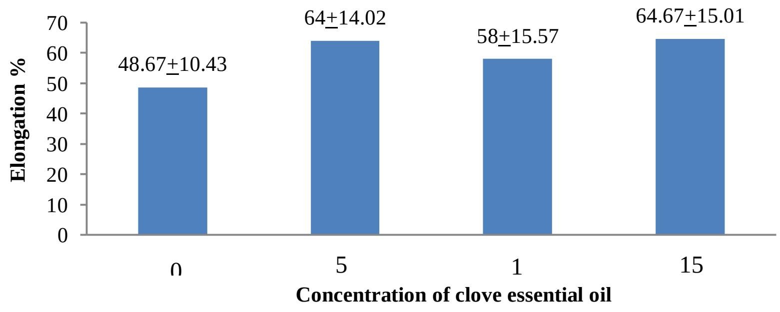

Figure 2: Elongation of whey-konjac film with the addition of clove essential oil

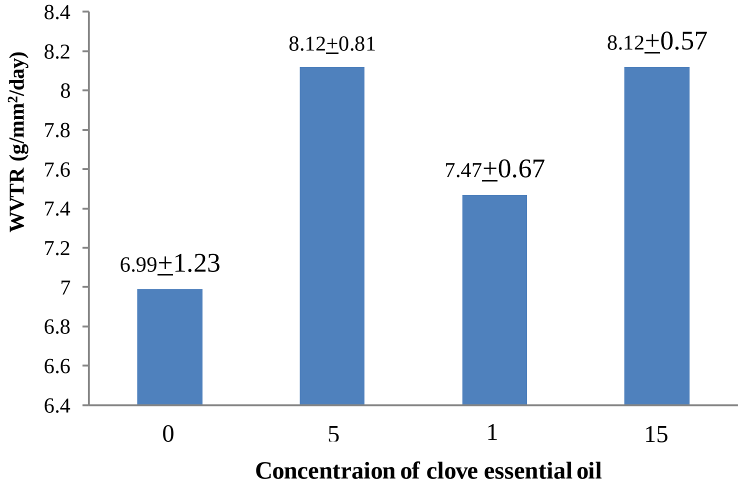

Figure 3: Water Vapor Transmission Rate (WVTR) of whey-konjac film with the addition clove essential oil

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structure and structural integrity (Shi et al., 2016). Xu et al. (2020) stated that addition of clove essential oil with a higher concentration could disrupt the matrix arrangement by forming intermolecular bonds, especially around the dispersed clove oil droplets. Research by Atarés et al. (2010) showed that the incorporation of clove essential oil affects the tensile strength depending on the interaction of the composite film matrix with oil added. The decreasing of tensile strengths and elongation from a film that caused by increasing level of clove essential oil uses (Yang et al., 2018). Pagno et al. (2016) observed that the tensile strength of films made from quinoa flour containing oregano oil decreased from 3.5 to 1.2 MPa. Ghasemlou et al. (2013) also reported that corn starch-based films containing *Mentha pulegium* oil showed a decreasing in tensile strength and Bof et al. (2016) reported that the addition of lemon essential oil to chitosan film resulted in a decreasing in tensile strength from 18.3 to 6.7 MPa. The tensile strength of the film also influenced by addition of glycerol plasticizer where it reduced the molecular tension between matrix in the film and then resulting in a weak edible film. The addition of glycerol also thought to reduce the binding force between starch molecules during evaporation, thereby reducing the film's strength to mechanical treatment (Fahrullah et al., 2020a). The presence of plasticizer reduced density of interactions between proteins, increased the mobility of polypeptide chain, and made the film less resistant and deformable (Silva et al., 2020). Overall, whey-konjac films formulated with different plasticizers of glycerol and clove essential oil had good tensile strength, indicating potential applications as edible packaging materials for various food products. Plasticizers add to polymers to overcome brittleness, provide flexibility, and increase toughness (Mekonnen et al., 2013). The increasing of mechanical characteristic of edible films by using plasticizer is very necessary (Dick et al., 2015).

The use of clove essential oil had no a significant difference ($p>0.05$) in the film elongations. The percentage of elongation resulted in this research was not yet standard which it must be at least 70% Japanese Industrial Standard (JIS). This elongation value was inversely proportional to the tensile strength resulting. This phenomenon probably occurs by the plasticizing effect of clove essential oil and deformability of oil droplets which increase mobility and elasticity of the polymer chains (Xu et al., 2020). Films were containing more concentration of clove essential oil where the oil droplets distributed in the whey-konjac composite films affect structure of the film network which affects to elongation. High elongation indicated that film was more flexible when subjected to physical stress. In this research, the elongation value was measured to explore effect of clove essential oil and glycerol plasticizer, from the results obtained, whey-konjac-based films showed a lower elongation values than research with similar materials. Research by Fahrullah et al. (2021b) using the same concentration of clove oil but different concentrations of glycerol showed that 30% increased the elongation of films to 53.21-78%. The tendency of hydrogen bonds between plasticizer molecules and matrix chains determined how strongly the polymer was connected to the plasticizer (Farhan and Hani, 2017). The plasticizer glycerol has an ability to absorb the high amounts of humidity and moisture in the making process of edible film (Dick et al., 2015). Glycerol was effective for reducing internal hydrogen bonds by creating intermolecular spaces and reducing film tightness and increasing its flexibility so that the addition of glycerol as a plasticizer results in good film elongation compared to sorbitol and polyethylene glycol (Fahrullah et al., 2020a). Therefore, providing more glycerol in the film-forming solution increases flexibility of the whey-konjac film but reduces its mechanical resistance (tensile strength). Overall, the whey-konjac film was added with clove essential oil gave a good elongation and indicated that this film had a potential as the packaging material with a good physical characteristics.

The highest WVTR that resulted from addition of 15% clove essential oil was 8.12 g/mm^2/day with ranges from 6 to 8 g/mm^2/day. This result already fit the standards of the JIS which requires a maximum of 10 g/mm^2/day. The best quality of edible film have a low WVTR value that give function to control the movement of water and it will be extend the storage of product (Xu et al., 2020). The whey-konjac polymer chain, which was opened by addition of clove essential oil in the film matrix, gave an increasing diffusibility of water vapor through the surface of film that caused an increasing rate of water vapor transmission (Nisar et al., 2018). This finding was different from the results of Teixeira et al. (2014) who observed that the WVTR of fish protein-based films tended to decrease when clove essential oil was added. Several researches using whey-konjac film as a base material produced various WVTR such as Fahrullah et al. (2020a) with 7.63-7.96 g/mm^2/day, Fahrullah et al. (2020b) with 7.64-8.45 g/mm^2/day, and Fahrullah et al. (2021b) with 7.15-8.61 g/mm^2/day. The increasing WVTR attributes to the hydrophilic nature of glycerol plasticizer. The concentration of glycerol affects to reorganization of polysaccharide network which allows water molecules to diffuse more easily and provides a higher WVTR (Khazaei et al., 2014). Yang et al. (2018) stated that the water vapor transfer mostly occurs through hydrophilic part of the film structure and generally depends on the hydrophilic ratio of the film components. An increasing rate of water vapor transmission with an increasing of

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hydrophilic plasticizers was common in edible films such as those formed from starch (Yan et al., 2012).

Microscopy technique is useful for knowing the microstructure of edible films on a micro scale. SEM is a technique that widely used in edible film research for microstructural characterization (Arzate-Vázquez et al., 2012; Fahrullah et al., 2020a, b, 2021a, b; Fahrullah, 2021; Fahrullah and Ervandi, 2021; Saravani Pak et al., 2020). Another important aspect related to microscopy technique is the level of magnification used to observe the film (Arzate-Vázquez et al., 2012). In this research, the addition of clove essential oil at different concentrations and magnification of 1,000 and 20,000 times produced the microstructure.

Microstructure of whey-konjac edible film with addition of 0% clove essential oil produced a structure with a flat surface; the mixture of whey solution with konjac was evenly distributed, characterized by the highest tensile strength value among other treatments. This flat surface structure occured because of method of making the film and the good mixing process. Cofelice et al. (2019) stated that the heating method, homogenization process, emulsion composition, and structural component at the end of drying process were determinant factors of microstructure of film. The heating process also resulted in fewer lumps and resulted in a flat and smooth surface structure with a magnification of 20,000 times. It is related to the physical characteristic of resulting film where the tensile strength increases and resistant to brittleness. Microstructure of edible film whey-konjac with addition 10 and 15% clove essential oil produced a less homogeneous surface structure. The whey protein matrix with konjac seemed to be disturbed by the presence of clove oil so that an emulsion process occurred. The level of clove essential oil, with more uses, causes an increase to interaction between oil particles and polymer matrix. This increase tends to produce or form large pores (Fahrullah et al., 2021b). Whey protein has a role to stabilization with forming a stabilizing layer around the fat droplets (Kurek et al., 2014). Whey protein has an ability to form a barrier around the oil droplets in migration process. The addition of 10 and 15% clove oil showed that there were mostly oil droplets on the surface of film with a magnification of 20,000 times. The aggregation process begins after the homogenization process and occurs during a drying process, so the formation of these oil droplets during drying can cause a less homogeneous surface structure of the film (Fahrullah et al., 2021a). Al-Hashimi et al. (2020) stated that addition of clove oil to the film is an important factor in barrier efficiency and their distribution in the film matrix greatly affect microstructure of the resulting film; this is in accordance with what the authors did in investigating of whey-based edible film characteristics. Konjac produced water vapor barrier characteristics that increased proportionally with addition of clove essential oil.

Conclusion

This research concluded that the use of clove essential oil with different concentrations had no effect on tensile strength, elongation, and WVTR, but the addition of clove essential oil had promising potential to improve the physical characteristic of whey-konjac edible films. The concentration of 5% clove essential oil resulted in the best physical properties of film.

Author contributions

F.F. and F.M. designed the study; F.F. conducted the experimental work; F.F. and A.N. analyzed the data; F.F., A.N., and F.M wrote the manuscript. All authors read and approved the final manuscript.

Conflicts of interest

All the authors declared that this is no conflict of interest in the study.

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