

# Characterization of corn starch-based edible film incorporated with nutmeg oil nanoemulsion

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**Abstract.** This study aimed to formulate corn starch-based edible films by varying concentrations of nutmeg oil nanoemulsion and glycerol. Furthermore, the resulted edible film was characterized by its mechanical properties and antibacterial activity. The edible films were made using corn starch, nutmeg oil nanoemulsion, and glycerol. Concentrations of nutmeg oil nanoemulsion were 1%, 2%, and 3%, and glycerol were 10%, 20%, and 30%. Results indicated that the increase of nutmeg oil nanoemulsion concentration could increase the film thickness. However, the nutmeg oil had no effect on the film tensile strength and elongation. Glycerol had no effect on the film tensile strength. The best treatment of the corn starch-based film was obtained by adding 1% of nutmeg oil and 30% of glycerol, yielding a tensile strength of 18.73 Kg/mm<sup>2</sup>, elongation of 69.44% and thickness of 0.0840. The addition of 1% nutmeg oil nanoemulsion has been able to inhibit the growth of two types of the bacteria tested (*Staphylococcus aureus* and *Escherichia coli*).

## 1. Introduction

Food products like fruits, vegetables, meat, and processed products are easily damaged and degraded. Therefore, the packaging is used to reduce food degradation. One of the primary packaging types is an edible film, a thin layer coating the food that can be consumed and naturally degraded [1]. Edible films are, in fact, a relatively new food preservation technique; yet, they have been widely researched. Several studies have shown that edible films can extend food shelf life and improve its quality.

One of the potential and widely researched polymer materials for edible films is starch. However, starch-based films have some weaknesses i.e. low resistance to water and low water vapor barrier due to its hydrophilic nature that affects its stability and mechanical properties [2]. However, the physical and functional characteristics of starch films can be improved by adding biopolymers or other materials that are hydrophobic and/or have antimicrobial properties such as essential oils and glycerol (plasticizers) [3].

Essential oils contain natural preservative compounds because of their antimicrobial properties [4]. These properties prevent microbial growth by interfering the microbial cytoplasmic membranes, disrupting cell constituents, and reacting with cell membranes; and thus, increasing permeability and causing cell component loss and/or coagulating cell contents [5]. Nutmeg oil is one of the essential



oils containing phytochemical compounds e.g. sabinene (25.4%),  $\alpha$ -pinene (15.8%) and myristicine (14.8%) [6].

The antibacterial compounds in nutmeg oil are non-polar, thus having a low solubility rate. Therefore, the nanoemulsion system is required to increase solubility, bio-accessibility, and bio-availability, and to maintain stability as well. The use of nanocomposites in edible film production could enhance the film tensile strength, storage modulus, glass transition temperature, and water vapor barrier properties [7].

This study used corn starch in making the edible films, reinforced with nutmeg oil nanoemulsion and glycerol. Corn starch contains a high amount of amylose, at approximately 25%, thus it can be utilized in developing the film matrix [8]. The addition of nutmeg oil nanoemulsion and glycerol into the edible film formulation could increase the mechanical and functional properties of the films. The aims of this study were to find the best formula out of several compositions of nutmeg oil nanoemulsion and glycerol in the edible films and to find out the effects of nutmeg oil nanoemulsion and glycerol addition on the film characteristics.

## 2. Materials and Methods

### 2.1. Materials

Materials used in this research were the essential oil of nutmeg oil (food grade) from PT. Djasula Wangi, Jakarta, Indonesia. Tween 80 (polyoxyethylene-20-sorbitan monooleate) and Glycerol analytical standard (Sigma-Aldrich, US). Corn starch was purchased from local market in Banda Aceh, Indonesia.

### 2.2. Preparation of Nutmeg Oil Nanoemulsions

Nanoemulsion was produced by using *High-Pressure Homogenization* (HPH) method. The type of emulsion was oil in water (O/W) with the nutmeg oil as the dispersed phase and water as the dispersing phase. The coarse emulsion was prepared by dispersing 15% of nutmeg oil into the double distilled water and tween 80 as a surfactant. The concentration of tween 80 was 20% of the nutmeg oil (w/v). The mixture then homogenized by a high shear homogenizer (IKA T25 digital Ultra Turrax) at 12000 rpm for 5 minutes. The coarse emulsion was then passed into High-Pressure Homogenizer (HPH) at 500 bar with 3 cycles [9].

### 2.3. Preparation of Edible Films

Edible films were prepared by dissolving 3% of corn starch and glycerol (10%, 20%, 30%) into the aqueous solution. The suspension was stirred at 85°C for 30 minutes. Afterward, the suspension was cooled to 45°C and then the nutmeg oil nanoemulsion at the concentration of 1%, 2%, 3% was added to the suspension. The mixture was cast onto 30 cm x 20 cm glass plates and was dried at  $\pm 50^\circ\text{C}$  for 12 hours and subsequently cooled at room temperature for 24 hours. Then the films could be easily removed out from the glass plates. The films were then analyzed [10].

### 2.4. Characterization of Edible Film

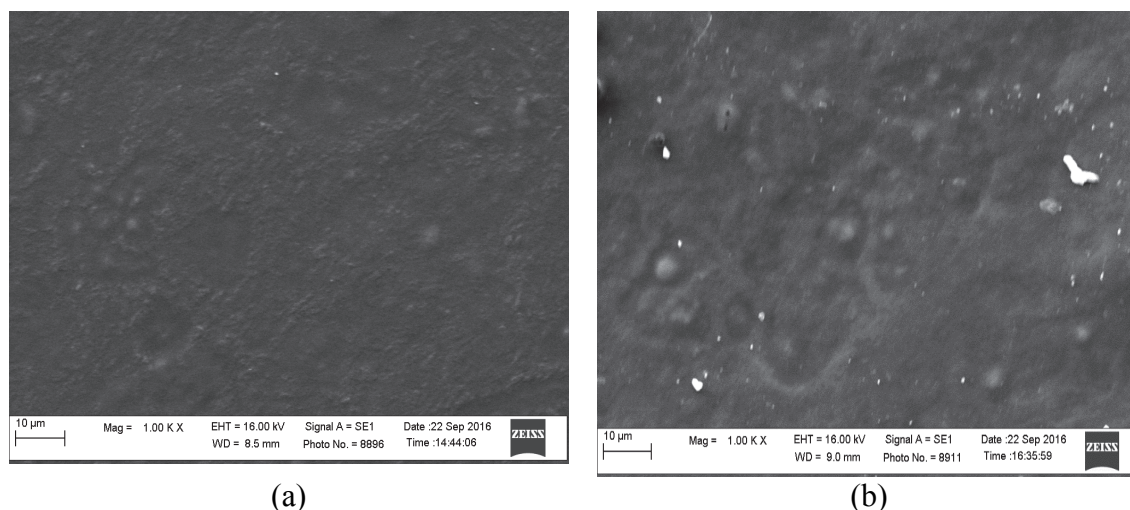
The characteristics of the resulted films were observed by measuring those mechanical properties; tensile strength, thickness, elongation [11]; and the antibacterial as well [12].

## 3. Results and Discussions

### 3.1. Morphology of edible film

The analysis results of the edible film surface morphology can be seen in Figure 1. Based on the SEM test results in Figure 1, It can be seen that the film morphology with no addition of nutmeg nanoemulsion, 20% of corn starch, and 30% of glycerol shows viscous, smooth, and non-porous surface of the film molecular structure (Figure 1a). On the other hand, the surface of the film

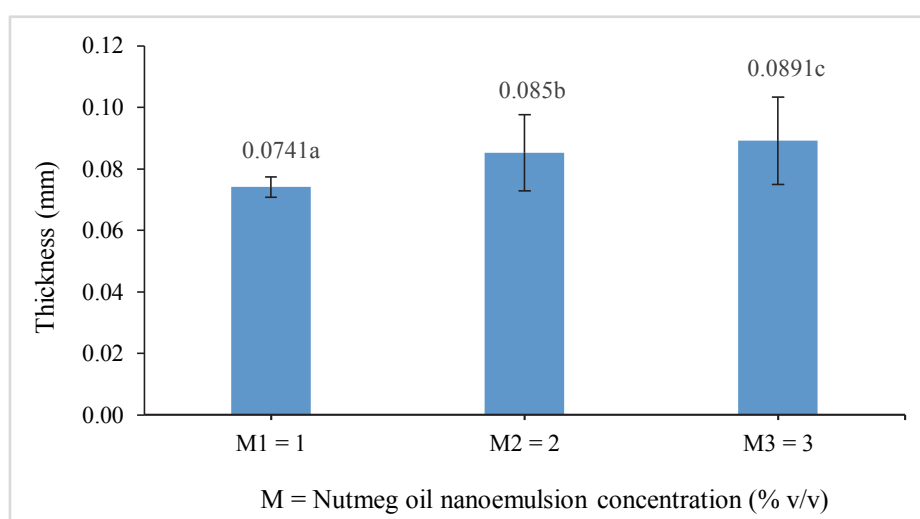
molecular structure produced by adding 1% of nutmeg oil nanoemulsion, 20% of corn starch, and 30% of glycerol is quite porous (Figure 1b).



**Figure 1.** The film surface with no addition of nutmeg oil nanoemulsion, 20% of starch, and 30% of glycerol (a) and with the addition of 1% nutmeg oil nanoemulsion, 20% starch, and 30% glycerol (b) using 100x magnification

### 3.2. Thickness (mm)

The film thickness was measured by using a screw micrometer at five random points with the area of 7x7 cm each. The average measurement data of the thickness were collected. The thickness data showed that the films had a range of thickness from 0.0643 – 0.1013 mm with an average of 0.0830 mm.

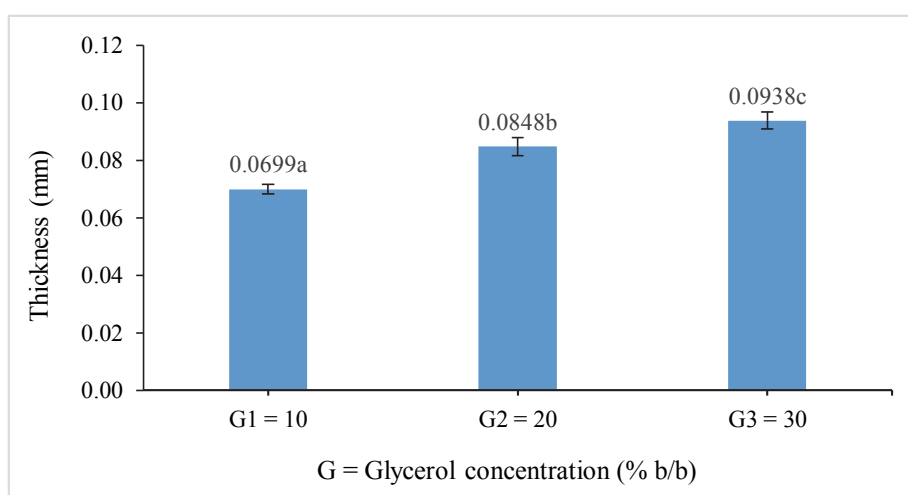


**Figure 2.** The effects of nutmeg oil nanoemulsion concentration (M) on edible film thickness

Figure 1 shows that nutmeg oil nanoemulsion at 3% of concentration had the highest thickness while at 1% of that resulted in the lowest thickness. It indicates that the higher the nutmeg oil concentration, the thicker the film. Then, after the LSD test between treatments was carried out, there was a

significant difference on the film thickness between the concentration of nutmeg oil nanoemulsion 1%, 2%, and 3%, shown from different notation. The nutmeg oil nanoemulsion consisted of 3 (three) components: nutmeg oil, water, and surfactant. The addition of nanoemulsion could enhance the viscosity of the solution leading an increase of the edible film thickness. The increase in thickness occurs due to differences in the concentration of the filmmaker, while the volume of solution poured on each plate is the same. Furthermore, both the total solid in the dried films and the polymers that make up the film matrix increased. Bertuzzi (2007) showed that the addition of glycerol concentration increased the viscosity of the solution thus increasing the film thickness as well. The increased concentration of the materials used will lead to an increase in film thickness (Mc Hugh, 1993).

Figure 2 shows that the glycerol concentration at 30% produced the highest thickness the edible film, while the glycerol concentration at 10% yielded the lowest thickness. It means that the higher of the glycerol concentration, the thicker of the films. Then, after the LSD test between treatments was carried out, there was a significant difference on the film thickness between the concentration of glycerol at 1%, 2%, and 3%, shown from different notation.



**Figure 3.** The effects of glycerol concentration (G) on edible film thickness

According to Zhang and Han [13], the film thickness elevates as the plasticizer increases, this is because glycerol can bind quite a lot of water, thereby raising viscosity. In addition, the viscosity of a solution has an impact on water evaporation in the drying process [14], thus, when the viscosity is higher, the film will be thicker.

### 3.3. Tensile Strength ( $\text{Kgf/mm}^2$ )

Tensile strength is the mechanical property of an edible film which reflects the maximum stress that the film sustains before it eventually breaks. Tensile strength shows the film strength in resisting the mechanical damage. The device used here was an autograph with the measurement in  $\text{Kgf/mm}^2$ . The value of tensile strength obtained in this study ranged from 15.07 – 18.93  $\text{Kgf/mm}^2$  with an average of 16.90  $\text{Kgf/mm}^2$ .

The addition of nanoemulsion at the concentration of 1%, 2%, 3% and glycerol of 10%, 20%, 30% was not notable in the film tensile strength. However, generally, it seems that the higher the concentration of nanoemulsion (M), the lower the film tensile strength, as depicted in Table 1. The decrease is mainly caused by the ability of glycerol to enhance flexibility and lessen intermolecular forces along the polymer chains [15].

**Table 1.** The effect of nutmeg oil concentration on the mechanical properties of the edible films

Concentration of nutmeg oil nanoemulsion	Tensile strength (Kgf/mm <sup>2</sup> )	Elongation at break (%)
1%	17.38	35.57
2%	16.56	33.71
3%	15.78	32.89

**Table 2.** The effect of glycerol concentration on the mechanical properties of the edible films

Concentration of glycerol	Tensile strength (Kgf/mm <sup>2</sup> )	Elongation at break (%)
10%	16.00	11.52
20%	16.67	28.93
30%	18.04	52.84

The decrease in tensile strength and increase in elongation are common results of essential-oil incorporation and have been broadly discussed in research into other biopolymer films. Maizura et al. (12) reported a decrease in the TS of the film prepared from a starch-alginate film to which different concentrations of lemongrass oil had been added. They reported that this was due to the effect of the lipid on the starch chains and that the phase rich in polysaccharide had a higher tensile strength than lipid phase. Furthermore, the addition of glycerol led to the formation of free volumes, a result of broken bonds between polysaccharides [16]. The tensile strength keeps declining as molecular interactions decreases [11]. Glycerol can penetrate into polysaccharide molecules and remove hydrogen bonds.

### 3.4. Elongation (%)

Elongation is the maximum extension percentage that an edible film can achieve before it is finally broken/torn. The observation data in this study showed that the elongation values obtained ranged from 4.81% to 69.44% with an average of 31.10%.

Table 2 shows that the largest percentage of elongation of 52.84% was obtained at 30% of glycerol concentration and the lowest of 11.52% was at 10% of glycerol concentration. This suggests that the increase of glycerol concentration tends to elevate the percentage of the film elongation. The increase of glycerol may decrease the intermolecular force of the edible film matrix structure, increase flexibility, and lower the number of hydrogen bonds; thus, reducing fragility and not easily breaking.

### 3.5. The antibacterial activity of edible films

The results of the study showed that the edible films with the addition of nutmeg oil nanoemulsion were able to inhibit the growth of *Staphylococcus aureus* and *Escherichia coli*. The film antimicrobial activity with 3% of nutmeg oil nanoemulsion addition was capable of inhibiting *Staphylococcus aureus* at 18.27 mm and *Escherichia coli* at 14.89 mm. Reinforcing nutmeg oil nanoemulsion to the films causes the obstruction of the growth of tested microbes (Table 3). When the nanoemulsion was added to the films, it would diffuse into the agar medium and produce clear zones in the microbial growth medium.

**Table 3.** The inhibition zone diameter of edible film with the addition nutmeg oil nanoemulsion against *S. aureus* and *E. coli*

Type of bacteria	Nutmeg oil nanoemulsion concentration	Inhibition zone (mm)
<i>Staphylococcus aureus</i>	1%	10.04
	2%	14.62
	3%	18.27
<i>Escherichia coli</i>	1%	10.76
	2%	12.22
	3%	14.89

The Table 3 describes that the higher of nutmeg oil nanoemulsion concentration, the greater the ability to inhibit the growth of tested bacteria. Factors affecting the size of the inhibitory area include the sensitivity of the organism, the culture medium, the incubation conditions, and the speed of the agar diffusion. Factors affecting the speed of agar diffusion are the concentration of microorganisms, the composition of the media, the incubation temperature, and the incubation time. According to [17], the diameter of the inhibitory zone does not always elevate in corresponding with the rise of the bacterial concentration. It is highly likely that different diameters of the inhibitory zones over a period occur due to the differences in the diffusion speed of antibacterial compounds on the agar medium and the different types and concentrations of antibacterial compounds.

It can be concluded that the addition of nutmeg oil nanoemulsion of 1% has been able to inhibit the growth of two types of the bacteria tested (*Staphylococcus aureus* and *Escherichia coli*). Therefore, the addition of 1% nutmeg oil nanoemulsion is chosen as the minimal concentration of the inhibitory ability.

#### 4. Conclusion

The concentrations of nutmeg oil nanoemulsion and glycerol have had an effect on the characterization of corn starch-based edible films. The best treatment of edible film was produced by adding 1% of nutmeg oil nanoemulsion and 30% of glycerol, resulting in the film tensile strength of 18,73 Kgf/mm<sup>2</sup>, elongation of 69.44% and thickness of 0.0840 mm. The addition of 1% nutmeg oil nanoemulsion was able to inhibit the growth of two bacteria tested (*Staphylococcus aureus* and *Escherichia coli*).

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