

## Physical and mechanical properties of agar based edible film with glycerol plasticizer

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### Abstract

Appropriate concentration of base material and plasticizer is required to obtain good physical and mechanical properties of edible film for food packaging and preservation functions. The aim of this study was to obtain the best combination of the base material and plasticizer in the manufacture of agar films based on physical and mechanical properties. Results showed that the physical and mechanical properties of the agar edible film were affected by the agar and glycerol concentrations. Increasing agar concentrations resulted in the increase in the film thickness, tensile strength (TS), and elongation at break (EAB), but decreased the film solubility. While increasing glycerol concentration tended to increase the film thickness and solubility, but decrease the TS of the film. The best concentration combination of agar and glycerol in this study was 3 and 10%, respectively.

### Keywords

Edible film

Agar

Glycerol

Plasticizer

Physical properties

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### Introduction

Edible films are thin layers made from edible materials which have been introduced as a barrier layer to improve consumer acceptability and shelf life of food products. The protective function of the edible films is to prevent oxidation processes, absorption and desorption of moisture, contamination, microbial growth and sensory changes. Edible films have the potential to maintain the quality of food products. Edible films can control moisture, oxygen, carbon dioxide, flavour, aroma displacement, and atmospheric conditions of foodstuffs (Pavlati and Orta, 2009; Avena-Bustillos and McHugh, 2012).

Edible films can be produced from materials with the film forming ability. During manufacturing, film materials must be dispersed and dissolved in a solvent such as water, alcohol or mixture of water and alcohol or a mixture of other solvents. Plasticizers, antimicrobial agents, colors or flavors can be added in this process (Bourtoom, 2008). Materials of edible films can be derived from polysaccharides, proteins, lipids, resins or composites with or without the addition of plasticizers (Cerqueira *et al.*, 2011). Some polysaccharides from seaweed extracts such as carrageenan, alginate and agar have been used as materials for manufacturing of edible films (Souza *et al.*, 2010a; Martins *et al.*, 2012; Galus and Lenart,

2013; Rhim, 2013; Abdou and Sorour, 2014; Shankar *et al.*, 2014; Rhim and Wang, 2014).

Agar is a hydrocolloid consisting of a mixture of agarose and agaropectin. Agar can form reversible gels simply by cooling and heating. It can form strong gels characterized by melting point far above the initial gelation temperature (Dhanapal *et al.*, 2012). The chemical structure of agar is mainly consisted of repeating units of D-galactose and 3,6-anhydro-L-galactose, with slight variations and low content of sulfate esters. Agar is also a mixture of polysaccharides composed of a combination of linear dextrose and levo galactose (Phan *et al.*, 2005; Stanley, 2006).

The use of agar as a base material of edible films has been studied and the results showed that agar films have good physical and mechanical properties namely transparent, clear, homogeneous, flexible, and easy to handle. Agar films also have good functional properties which make it suitable to be used as food packaging and coating material (Lacroix and Tien, 2005; Phan *et al.*, 2009). The aim of this study was to obtain the best combination of the base material and plasticizer in the manufacture of agar films based on physical and mechanical properties.

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## Materials and Methods

### Materials

Commercial agar powder (gel strength 900±40 g/cm<sup>2</sup>, particle size 80 mesh, moisture 20%, and yellowish color) was purchased from Golden Agar Sentosa (Surabaya, Indonesia). Other reagents for films formulations i.e. glycerol (food grade) and distilled water were obtained from local chemical store.

### Film preparation

Films were prepared according to the method of Sousa *et al.* (2010a) with a slight modification. Agar powder was dissolved in distilled water at 95°C for at least 30 min under stirring. The concentration of agar solution was 1, 2, and 3% (w/v). Glycerol was then added to the agar solution at a concentration of 5, 10, and 15% (w/w). The solution was kept at 95°C and stirred for 10 min after which the solutions were cooled down to 75°C prior to casted in petri dishes (10 cm diameters). The solution provided 0.20 ml/cm<sup>2</sup> and was evenly spread over the dishes. The dishes were placed on leveled surfaces to obtain films of homogeneous thickness, and subsequently dried in an oven at 50°C for 24 h. Dried films were peeled off from the dishes and stored in desiccators at room temperature.

### Thickness

The thickness of the films was measured using a digital micrometer with an accuracy of ±1 µm (Krisbow KW06-85). Nine thickness measurements were randomly taken on each film sample, and a mean value was used in the calculation.

### Solubility

The solubility of film samples was determined according to the method of Ahmad *et al.* (2012). Solubility test was done using film samples of 3 x 2 cm in size. Samples were dried at 105°C for 24 h and weighed (W1). Each sample was then inserted into a 50 ml centrifuge tube containing 10 ml of distilled water. Samples were stored for 24 h at room temperature and stirred slowly on a periodic basis using a shaker. The solution was filtered, and the residues remained on the filter paper were dried in an oven at 105°C for 24 h after which the samples were weighed to determine the dry matter soluble in water (W2). Solubility was calculated using the formula:

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

### Tensile strength (TS) and elongation at break (EAB)

TS and EAB were measured using modified digital fruit sclerometer. Film strips (35 x 50 mm) were mounted to a grip of digital fruit sclerometer. The maximum force required to rupture each film was read from the digital display of the device. TS was calculated by dividing the maximum force to rupture (F) and the cross-section area of the films (A), which was obtained by multiplying the width and the average thickness of the film strips. TS was calculated using the following formula:

$$TS \text{ (MPa)} = \frac{F}{A} \quad (2)$$

EAB was calculated by dividing the increase in length of the film strips when the rupture occurred (b) with the initial length of the film strips before loading (a). EAB was calculated using the formula:

$$EAB \text{ (\%)} = \frac{b}{a} \times 100 \quad (3)$$

### Experimental design and statistical analysis

The experiment was conducted using full factorial design with three replications. The concentration treatments used in the study were 1, 2, 3% (w/v) agar and 5, 10, 15% (w/w) glycerol. The effects of the treatments were analyzed using ANOVA, and the difference between treatment effects was tested using Duncan's multiple range test. Significant difference was considered at 95% probability. Data analysis was performed using SPSS software (SPSS Inc.).

## Results and Discussion

### Film thickness

The films thickness is an important characteristic in determining the feasibility of edible films as packaging materials for food products since the thickness of the films affects other characteristics of the films, such as tensile strength, elongation, and water vapor permeability (McHugh *et al.*, 1994; Galus and Lenart, 2013). The films thickness is dependent on both film composition and processing parameters (Garcia *et al.*, 2009).

The results of ANOVA (Table 2) indicated that agar and glycerol concentration, and their interaction significantly affected films thickness ( $p < 0,05$ ). Increasing the agar concentration provides more dissolved solids in solution resulting in thicker films. Increased films thickness due to the base material effect can be related to the compound's unique colloidal features such as thickening and suspending agent, and the interaction between components (Galus and Lenart, 2013). The effect of increasing

Table 1. Physical and mechanical properties of edible films

Agar (%w/v)	Glycerol (%w/w)	Thickness ( $\mu\text{m}$ )	Solubility (%)	TS (MPa)	EAB (%)
1	5	31.2 $\pm$ 0.7	68.1 $\pm$ 4.8	19.8 $\pm$ 6.2	16.33 $\pm$ 2.1
	10	34.4 $\pm$ 2.4	69.9 $\pm$ 4.7	21.0 $\pm$ 6.8	14.33 $\pm$ 3.8
	15	39.1 $\pm$ 1.5	75.3 $\pm$ 8.8	20.2 $\pm$ 8.0	15.33 $\pm$ 3.5
2	5	47.5 $\pm$ 1.5	59.6 $\pm$ 5.1	35.3 $\pm$ 9.6	23.33 $\pm$ 2.5
	10	46.9 $\pm$ 1.9	62.1 $\pm$ 8.5	30.5 $\pm$ 2.3	21.67 $\pm$ 2.5
	15	47.9 $\pm$ 2.7	69.3 $\pm$ 4.0	25.3 $\pm$ 4.9	22.67 $\pm$ 2.1
3	5	57.0 $\pm$ 6.3	49.9 $\pm$ 1.0	33.9 $\pm$ 4.6	24.83 $\pm$ 2.3
	10	64.0 $\pm$ 3.2	53.8 $\pm$ 2.1	32.3 $\pm$ 5.8	31.78 $\pm$ 4.1
	15	69.6 $\pm$ 2.3	58.5 $\pm$ 3.3	25.2 $\pm$ 5.5	26.67 $\pm$ 1.5

Values were mean  $\pm$  standard deviation.

Table 2. Anova of agar and glycerol concentration based on physical and mechanical properties of edible films

Variable	Source	Mean Square	F	Sig.
Thickness	Agar	1860.0	217.1	0.0
	Glycerol	110.1	12.9	0.0
	Agar * Glycerol	28.9	3.4	0.0
Solubility	Agar	658.6	23.5	0.0
	Glycerol	171.1	6.1	0.0
	Agar * Glycerol	2.1	0.1	1.0
TS	Agar	303.4	7.7	0.0
	Glycerol	89.3	2.3	0.1
	Agar * Glycerol	25.9	0.7	0.6
EAB	Agar	350.4	43.5	0.0
	Glycerol	3.4	0.4	0.7
	Agar * Glycerol	20.3	2.5	0.1

concentrations of the base material on the increase of the films thickness observed in this study is in accordance with the results of previous studies reported by Kokoszka *et al.* (2010) on soy protein isolate-based edible films with glycerol plasticizer, and Song *et al.* (2012) on barley bran protein-gelatin composite films with sorbitol plasticizer.

The addition of glycerol concentration in the manufacture of edible film also resulted in increasing film thickness. This observation is consistent with Sudaryati *et al.* (2010) that improvement of thickness due to the addition of glycerol is because the glycerol molecules will occupy voids in the matrix and interact with edible film forming polymer, which causes the distance between the polymer increases thus improving the film thickness. Bourtoom (2007) has reported that the plasticizer added to the manufacture of edible film can bind with starch to form starch-plasticizer polymer, so that the starch-starch bond is replaced by the starch-glycerol-starch bond which led to improvement of the film thickness. Nemet *et al.* (2010) have reported that the film-forming solution with higher concentrations of glycerol has a higher dry matter content resulting in a thicker film. Furthermore, high glycerol concentration can increase the ability to absorb moisture to some extent

and can cause an increase in films thickness due to swelling process (Vieira *et al.*, 2011; Ahmadi *et al.*, 2012).

The films thickness resulted in this study ranges between 31.2-69.6  $\mu\text{m}$  depending on the concentration of agar and glycerol (Table 1). Similar results have been reported by several researchers using agar (Phan *et al.*, 2005; Kanmani and Rhim, 2014), carrageenan and locust bean gum (Martins *et al.*, 2012). Lower film thickness (12.5-26.8  $\mu\text{m}$ ) was reported by Galus and Lenart (2013) using sodium alginate and pectin.

#### Solubility

Solubility is a physical property related to the ability of edible films to dissolve in water, so that when ingested it can be digested properly, or if discharged into the environment, it can decompose naturally. Soluble-film packaging is convenient to use in ready to eat food products as it melts in boiled water or in the consumer's mouth (Pitak and Rakshit, 2011). On the other hand, there is a need for an edible film that has low solubility to enhance product integrity and water resistance (Perez-Gago and Krochta, 1999). Therefore, the films solubility is tailored to the needs of the user since high solubility

Table 3. Main Factor Analysis Using Duncan's Multiple Range Test

Treatment	Thickness ( $\mu\text{m}$ )	Solubility (%)	TS (MPa)	EAB (%)
<b>Agar (%w/v)</b>				
1	34.9 $\pm$ 3.7 <sup>a</sup>	71.1 $\pm$ 6.4 <sup>a</sup>	20.4 $\pm$ 6.1 <sup>a</sup>	15.3 $\pm$ 2.9 <sup>a</sup>
2	47.4 $\pm$ 1.9 <sup>b</sup>	63.7 $\pm$ 6.9 <sup>b</sup>	30.4 $\pm$ 7.0 <sup>b</sup>	22.6 $\pm$ 2.2 <sup>b</sup>
3	63.6 $\pm$ 6.6 <sup>c</sup>	54.1 $\pm$ 4.2 <sup>c</sup>	30.4 $\pm$ 6.1 <sup>b</sup>	27.8 $\pm$ 4.0 <sup>c</sup>
<b>Glycerol (%w/w)</b>				
5	45.2 $\pm$ 11.8 <sup>A</sup>	59.2 $\pm$ 8.7 <sup>A</sup>	29.7 $\pm$ 9.6 <sup>A</sup>	21.5 $\pm$ 4.4 <sup>A</sup>
10	48.4 $\pm$ 13.1 <sup>B</sup>	61.9 $\pm$ 8.6 <sup>A</sup>	27.9 $\pm$ 7.0 <sup>A</sup>	22.6 $\pm$ 8.2 <sup>A</sup>
15	52.2 $\pm$ 13.8 <sup>C</sup>	67.7 $\pm$ 9.0 <sup>B</sup>	23.6 $\pm$ 6.0 <sup>A</sup>	21.6 $\pm$ 5.4 <sup>A</sup>

Values were mean  $\pm$  standard deviation. Values with the same superscript letters within a column were not significantly different ( $p > 0.05$ )

can not protect the product from humidity and water loss (Gontard *et al.*, 1993).

The result of ANOVA in Table 2 indicated that concentration of agar and glycerol affected the film solubility ( $p < 0.05$ ). Increased concentration of agar was inversely affecting the films solubility (Table 3). This is caused by the increased content of insoluble solids from the agar and the increasing number of bonds between molecules in a solution of the edible film. In addition, the hydrophilic nature of the agar and only soluble in hot water solvent (Dhanapal *et al.*, 2012). Whereas the increase in glycerol concentration resulted in increasing the film solubility. This is in accordance with the report of Farahnaky *et al.* (2013) in the manufacture of wheat starch edible film, and Wittaya (2013) in the manufacture of edible film from mung bean.

Improvement of the film solubility due to the influence of increasing concentrations of glycerol caused by hydrophilic and hygroscopic properties of glycerol. Bourtoom (2008) has reported that the increase of the films solubility with increasing plasticizer concentrations can be explained by the hydrophilic properties of plasticizer, where the dry matter solubilized in water is likely formed by the plasticizer. Therefore, the increase in the glycerol content of edible film will increase the content of dry matter solubilized in water, thereby increasing the films solubility. Ramos *et al.* (2013) suggested that the improve of films solubility because of the increase concentration of glycerol is caused by hygroscopic properties of glycerol which attract and bind water molecules, thus supporting the film surface wetting and moisture absorption. The high solubility of the film with glycerol plasticizer is because the glycerol has a strong affinity to water molecules, as well as low molecular weight facilitates the entry of glycerol between polymer chains, thereby increasing the free space volume between the chain (Sanyang *et al.*,

2015).

The results showed that the highest solubility of edible films was obtained from combination of 1% agar and 15% glycerol (Table 1). Solubility of the edible films produced was relatively high ranging from 49.9-75.4%, whereas the solubility of protein-based edible films ranged from 40.8-47.7% (Blanco-Pascual *et al.*, 2013), and chitosan-based edible films ranged from 42.1-47.1% (Bourbon *et al.*, 2011). Comparable solubility values (40.9-64.2%) were reported by Pitak and Rakshit (2011) on banana flour and chitosan composite films.

#### Tensile strength (TS)

Edible films as food packaging function to protect food during handling, transportation and marketing require high TS (Sousa *et al.*, 2010b; Pitak and Rakshit, 2011). The result of ANOVA in Table 2 indicated that the treatment of agar concentrations significantly affected the TS, while the glycerol concentration was not significant ( $P > 0.05$ ).

TS increased with the addition of agar concentration (Table 1). This is due to the addition of agar concentration in solution provides more affinity for higher number of hydrogen bonds formed on the edible film. Wu *et al.* (2009) reported that increasing the concentration of agar in the manufacture of potato starch based films caused TS to increase due to the formation of hydrogen bonds between starch and agar molecules, and yielded compact structure of the films. In addition, the increased agar concentration can produce dense films thus increasing the TS. Wu *et al.* (2009) reported that agar is contrast to other polysaccharides, where it could form a three-dimensional network gel which could result in dense film when the moisture vaporized. Increasing the TS value due to the effect of the increase in the base material concentration has also been reported in manufacture of protein and starch based films (Song

et al., 2012; Jongjareonrak et al., 2006; Al-Hassan and Norziah, 2012; Polnaya et al., 2012).

TS tended to decrease with increasing of glycerol concentration. The trend observed in this study was also observed by Venugopal (2011) that the addition of glycerol in the manufacture of edible film can reduce TS due to a decrease in the interaction between water molecules and agar. In addition, the increase in plasticizer concentrations increased the moisture content of the film because of its high hygroscopic nature, which also contributes to the reduction of the forces between the adjacent macromolecules (Sobral et al., 2001). Similarly, Liu et al. (2013) reported that increasing the glycerol concentration in the manufacture of starch-chitosan films tend to lower the TS value. The phenomenon of a decrease in TS with increase in plasticizer concentration has been reported by several other researchers (Saremnezhad et al., 2011; Arrieta et al., 2013; Wiset et al., 2014).

#### *Elongation at break (EAB)*

EAB is the ability of the films to extend before breaking. It describes the nature of the film plasticity. Plasticity or extensibility is generally required for a film to maintain its integrity when applied to food products (Galus and Lenart, 2013; Martins et al., 2012).

The results of ANOVA in Table 2 showed that agar concentration significantly affected the EAB ( $p < 0.05$ ), whereas the glycerol concentration and interactions between agar and glycerol concentrations were not significant ( $p > 0.05$ ). The EAB increased with increasing the concentration of agar (Table 1). Increasing agar concentration in the film solution will increase the water holding capacity thereby enhancing EAB. This is consistent with the report of Murdinah et al. (2007) that increasing alginate concentrations in the manufacture of film resulted in a higher water holding capacity producing better gel matrix that can increase the elongation of the film. Similarly Irianto et al. (2006) have reported that higher concentration of hydrocolloid carrageenan will increase the film elongation. Concentration, type of base material, and solvent used in the manufacture of film affect the elongation and tensile strength of the film (Herliany et al., 2013). Furthermore, it was reported that an increase of carrageenan concentration to 1.5% tended to increase the elongation of carrageenan-based films. Galus and Lenart (2013) reported that the increase in the proportion of alginate to 50% in the manufacture of composite films based on sodium alginate and pectin tended to increase the EAB and TS value. A similar result was obtained by Abdou and Sorour (2014) in their study on carrageenan

content in the manufacture of starch and carrageenan composite films.

The highest EAB was obtained from 3% agar and 10% glycerol (Table 3). Increasing glycerol concentrations up to 10% tends to increase the EAB due to reduction of the intermolecular interactions between the polymer chains that have an impact on increasing the elongation and flexibility of the film (Zhong and Xia, 2008; Kokoszka et al., 2010). Increase the amount of glycerol will reduce the strength of intermolecular forces that enhance mobility between molecular chains and lead to an increase in the elongation (Katili et al., 2013). Oses et al. (2009) reported that the increased content of plasticizer up to a certain concentration will increase the elongation; for the film with glycerol plasticizer, increasing the elongation occurred only at concentration of 40%. Similarly Liu et al. (2013) have reported that the addition of glycerol at 5% (w/w) and higher concentrations for starch-chitosan film resulted higher EAB values due to plasticization. The addition of glycerol promoted the interactions among chitosan, starch and glycerol through hydrogen bonding in films.

#### **Conclusions**

Physical and mechanical properties of edible films were highly dependent on the agar and glycerol concentration. Increasing concentrations of agar increase the film thickness, TS, and EAB, but decrease the solubility, while increasing glycerol concentrations tend to increase the thickness and solubility, but decrease the TS of the films. The best combination of the agar and glycerol concentration in this study was 3 and 10%, respectively.

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