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Edible film cassava starch/eggshell powder composite containing antioxidant: preparation and characterization

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Abstract. Edible film is a thin layer that is edible and acts as a protective food from environmental affect such as water vapor, oxygen, and moisture. Edible film with antioxidant content could prevent food from bacterial activity. In the present research, source of antioxidant is derived from “Kelakai” extract. This aims of this study are to evaluate the effect of eggshell powder concentration and cassava starch as raw material for edible film production and to study the physical and chemical properties of the edible film. The edible film was made from a mixture of eggshell powder (5%, 10%, 15%, 20%, 30%, wt/v) and cassava starch (5% wt/v) in distilled water. The mixture was added 1.5% v/v of glycerol and 5% and 10%, v/v of Kelakai (*Stenochlaena palustris*) extract. The mixture was heated up to ~96 °C, 100 rpm for 40 min. Then, mixture was poured into the acrylic mold (20x10 cm) and dried in an oven at 50 °C for 24 h. The optimum composition of edible film is 10% of eggshell powder which has thickness of 0.122 mm, 1.6 MPa of tensile strength, 25.4% of elongation. The surface morphology of edible film was shown the rough surface and transparent which contains of C-H, O-H, and C=O groups that identified by Fourier Transform Infra-Red (FT-IR) analysis. The antioxidant activity of edible film was shown by scavenging activity about 10.13% after 10 min.

1. Introduction

One of the obstacles ² food industries is the limited shelf life of the product. To overcome this issue edible film is used. Edible film is a thin layer with material that can be consumed and acts as a protector against external influences such as water vapour, oxygen, and moisture [1]. Edible films function as protective substances such as antimicrobials, antioxidants, flavors, and dyes that can improve the functionality of packaging materials by adding extracts from certain ingredients [2]. Edible films that made from natural resources have great potential due to biodegradability, biocompatibility, edibility and good application. The development of materials from natural materials continues to be developed, especially a combination of materials that can provide a synergy effect on the product.

Cassava (*Manihot esculenta crantz*) starch or tapioca is one of cheap ³st edible film's raw materials. Cassava starch consists of amylose and amylopectin which containing linear polymer with a molecular weight of about 10⁶ and a highly branched polymer with high molecular weight. The main component for film formation from cassava is the polymerization is amylose. However, the cass ⁵a starch film brittle and weak leading to inadequate mechanical properties. In order to improve the mechanical



properties of the film can be accomplished by adding plasticizers such as water, glycerol, sorbitol, and other low-molecular weight-polyhydroxy compounds. Glycerol and sorbitol are widely used as plasticizers because of their stability and edibility. Addition of plasticizers makes the brittle films more flexible, but also less strong. This problem has led to the development of mechanical properties of cassava starch film [3-6]. Some modification technique including composite material has been developed to improve the quality of edible film including addition of natural antioxidant on edible film [2, 7-9].

Eggshell contains calcium and has the potential as a material for fertilizer, animal feed, coating pigment, catalyst, and adsorbent. Eggshell as polymer composite is cheap, abundant, environmentally friendly, and a renewable resource. Research on edible film made from cassava starch, glycerol and chicken eggshell powder as fillers in the development of bio-fillers for thermoplastic starches, so that edible film improve compatibility and biodegradable by microorganisms [10]. In addition, researchers are used chitosan and oil for edible film functionalization which have antibacterial and antioxidant properties [1, 7, 11-13]. The development of edible film using the main waste products for eggshell and cassava starch as a constituent of edible film has been studied. However, study of influence of eggshell concentration in formation of edible films is limited. In this work, the effects of eggshell concentrations as filling material on edible film formation were studied. The functionalization of edible using Kelakai extract as natural plant in South Kalimantan was used for antioxidant resource. Furthermore, the physical and chemical properties of the edible film such as thickness, tensile strength, elongation, surface morphology, functional groups, and antioxidant activity were investigated.

2. Materials and Method

2.1. Materials

Cassava starch or tapioca (Brand "Pa Tani") was purchased from local market and eggshell was collected from bakery at Banjarbaru, Indonesia. Kelakai was collected from Gambut area, South Kalimantan, Indonesia. Glycerol (approx. 96%, $C_3H_8O_3$), 1,1-diphenyl-2-picrylhydrazil (DPPH), methanol (95%, CH_3OH) were purchased from Sigma Aldrich without further purification.

2.2. Preparation of Kelakai extract

Young Kelakai leaf was cleaned and dried for 48 h in an oven at 50 °C. Dried Kelakai leaves were blended and the powder was sieved at 40 mesh. A 10 g of Kelakai powder was extracted with 100 mL of aquadest heated-up at 50 °C and stirred for 60 min. The Kelakai extract (KE) filtrate was collected by filtration.

2.3. Preparation of edible film

Eggshell powder (EP) was made based on previous research [10]. Briefly, eggshell was washed and boiled for 15 min, then dried at 60 °C for 2 h. The eggshells were crushed and sieved at 40 mesh. The mixture of eggshell powder (5%, 10%, 15%, 20%, 30%, wt/v) and cassava starch (5% wt/v) was poured in a beaker glass. Then the mixture was added distilled water (93.5% v/v), 5% v/v of Kelakai extract and glycerol (1.5%, v/v). The mixture was then heated up to ± 96 °C with constant stirring of 100 rpm for 40 min. The mixture was then poured into a 20x10 cm acrylic mould and dried in an oven for 24 h at 50 °C.

2.4. Characterization and analysis

X-Ray Fluorescence (XRF) was used to identify component in the eggshell. The investigation of structure morphology of edible film was used Field-Emission Scanning Electron Microscopy (FE-SEM, JOEL JSM-6500F) with energy-dispersive X-ray spectroscopy (EDAX). Fourier Transform Infra-Red spectrometry (Bio-rad, Digilab FTS-3500) was used for identified the surface functional groups on sample. The thickness of film was measure by micrometer screw which takes 5 different locations on the edible film. Torsee's Electronic System Universal Testing Machine with rate 20

mm/min and loading 100 kg was used to calculate tensile strength and elongation at break of edible film. The brightness of the edible film was measured by taking an image of an object covered with the edible film.

3. Results and Discussion

The eggshell powder produced from local resource contained 98.81% of calcium, higher than other eggshell from literature at 95% [10]. The difference concentration of component depends on food consumed by chicken.

3.1. Characterization of edible film

The increasing concentration of eggshell powder increase the thickness of the edible film produced. This is because high concentration of eggshell powder as a filling material will create matrix which tight and thickened the edible film [10]. In the present research, the thickness was below the maximum edible film standard set by Japanese Industrial Standard (JIS) which is 0.25 mm. The detail data of thickness is shown in Table 1.

Table 1. Thickness of edible film

No	Concentration of eggshell powder (%)	Thickness (mm)
1	0	0.174 ± 0.0114
2	5	0.176 ± 0.0148
3	10	0.184 ± 0.0195
4	15	0.180 ± 0.0212
5	20	0.194 ± 0.0245
6	30	0.200 ± 0.0335

The eggshell powder affects the edible film brightness of and mechanical properties. As shown in Figure 1, the more concentration of eggshell powder added, the brightness decreased.

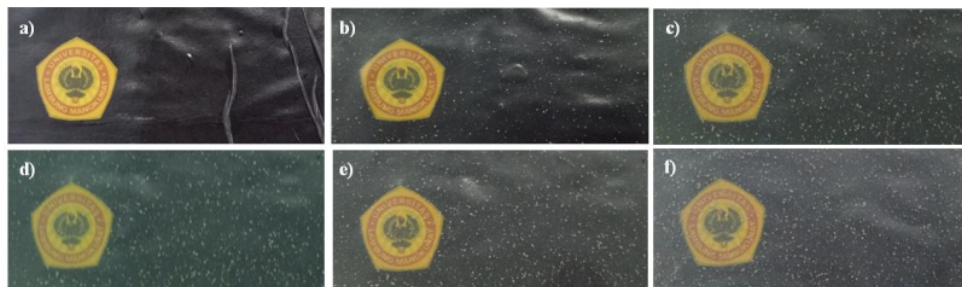


Figure 1. Edible film photographs with variation of eggshell powder concentration (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%; (f) 30%

Analysis of tensile strength and length of elongation test on edible film is shown in Figure 2. Tensile strength is test to determine the effect of the concentration of eggshell powder. Figure 2a shows the profile of tensile test values at various concentrations, the tensile strength on the sample composition of eggshell powder for 0%, 5%, 10%, 15%, 20%, 30% (wt/v) about 0.7; 1; 1.5; 1.2; 1; 0.9 MPa, respectively. The best the tensile strength was at of concentration 10% (1.5 MPa). The addition of eggshell powder as filler matrix in the edible structure gives optimum value. Interestingly, at the when concentrations of eggshell increased, the tensile strength decreased because it affects in the interconnection of structure in the matrix. Another mechanical property was observed for elongation at

break elongation on edible film, as shown in Figure 2b. The addition of eggshell powder has the effect of prolonging the greater termination, but at certain concentrations the value will decrease because too much filling material in the matrix resulting in decreased in flexibility and elasticity of edible film. This also applies to the tensile strength test which shows the same profile. The concentration 10% eggshell powder gave optimum value of elongation at break of 35%.

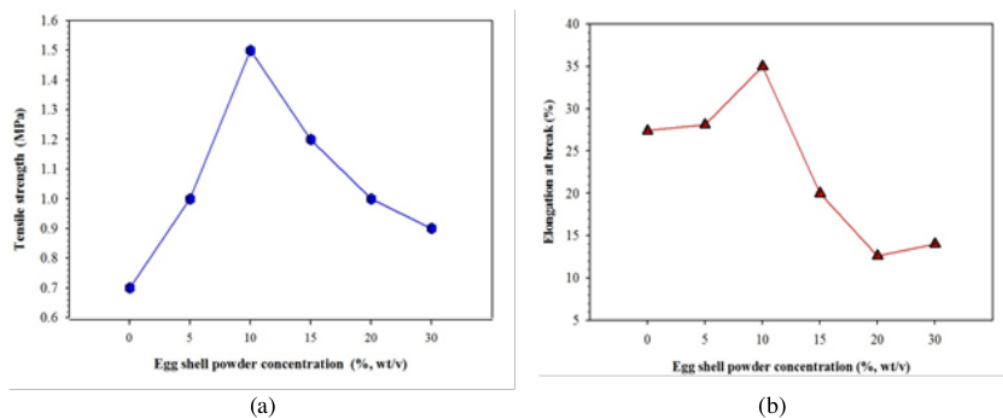


Figure 2. Effect of eggshell powder concentration (0%, 5%, 10%, 15%; 20%, 30%, wt/v) on: (a) tensile strength and (b) elongation at break of edible film

3.2. Addition of Kelakai extract in the edible film

The 10% of eggshell powder was used for further research for edible film in the presence of Kelakai extract (5% and 10%, v/v) antioxidants properties. However, the Kelakai extract affected the colour and mechanical properties of the edible film. The higher concentration of Kelakai extract, the yellowish the edible film. This is due to the effect of the original brown colour of the extract. Morphological structure of edible film observations with SEM are shown in Figure 3.

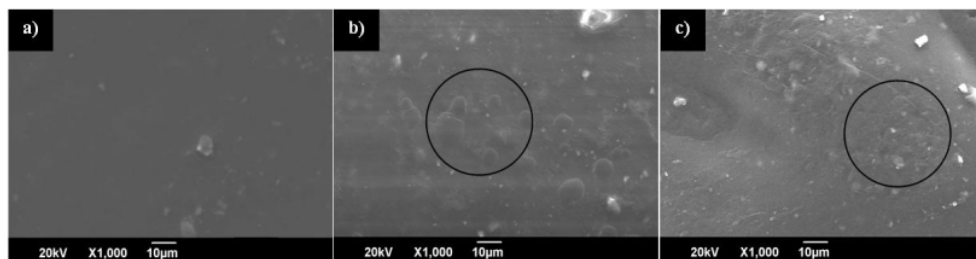


Figure 3. SEM images of edible film with Kelakai extract concentration of (a) 0%; (b) 5%; (c) 10% (v/v)

SEM observations showed that the addition of Kelakai extract filled the constituent matrix along with eggshell powder. Figure 3 shows rough surface and appear agglomerate become more by increasing extract concentration (circle in the figure). In addition, the presence of Kelakai extract affects the physical properties of edible film which can be seen in Table 2.

Table 2. Physical and mechanical properties of edible film in the presence of Kelakai extract

No	Kelakai extract concentration (%, v/v)	Thickness (mm)	Tensile strength (MPa)	Elongation at break (%)
1	0	0.184 ± 0.0167	1.5	35.0
2	5.0	0.174 ± 0.0114	1.5	27.4
3	10.0	0.122 ± 0.0084	1.6	25.4

The higher of extract concentration, the thinner edible is produced and tends to be difficult to form films. This is because antioxidants can enhance the hydrophobic nature of the film surface due to the small amount of carnosonic acid that has migrated to the surface during the drying process [14]. Tensile strength test results did not show a significant change, it means the addition of Kelakai extract did not affect the tensile strength. While the elongation test at termination has decreased slightly because there is a vacancy space in between the formed matrices (seen in Figure 3 (b) and 3 (c)) marked by the presence of agglomerate on the edible, this results in reduced elasticity of edible film.

FT-IR analysis was performed for the analysis of functional groups contained in resulting edible film is shown in Figure 4. The change in functional groups due to the effect of the composition of the extracts used (5% and 10%, v/v). The chemical structure of edible films is composed of three functional groups including O-H, C-H, C=O, C=C, and C-O groups. The -OH group is 3400 cm^{-1} , C-H group is 2927 cm^{-1} , group C=O at wave number 1714 cm^{-1} , group C=C at wave number 1497 cm^{-1} , and C-O group at 1027 cm^{-1} . A large area around C-H indicates the presence of CaCO_3 in eggshell powder, it shows the presence of calcite in the structural eggshell powder which is hygroscopic and moist [15].

Kelakai extract has a high absorbance at position O-H and C=O, the peak appeared when in edible film containing Kelakai extract. Edible film carrying extract showed signals that corresponded to the aromatic ring stretch C=C, this can be attributed to the presence of extracts that interact with the glycerol-starch matrix result to the presence of phenolic compounds from the extract at 1515 cm^{-1} . Other peaks around 1714 cm^{-1} was also detected and associated with the presence of C=O groups from carboxylic acids. Edible film which have extract show a greater reduction in the amount of -OH compared to without extract in changes in the availability of O-H in the polymer network [14].

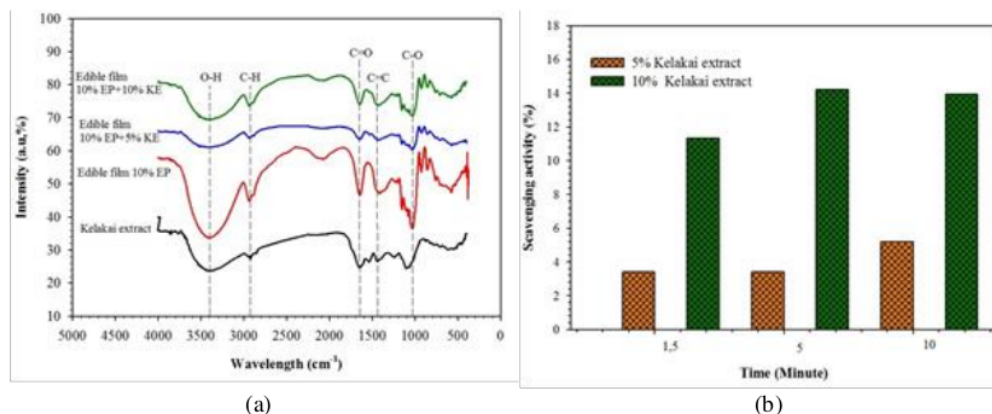


Figure 4. (a) FT-IR spectra of Kelakai extract, edible film and edible film containing Kelakai extract, and (b) Scavenging activity of edible film containing Kelakai extract (5% and 10%, v/v)

In order to evaluate the antioxidant activity, DPPH reagent was used to determine the efficiency of free radical capture by edible film, in terms of reduced purple colour solution. The reducing colour

was controlled up to 10 min. The scavenging activity of edible film is shown in Figure 4b. The containing Kelakai extracts of 5% and 10%, v/v are shown the activity about 5.19% and 13.93%, respectively. In the same condition, the scavenging activity of original Kelakai extract is 58.61%. In order word, the prepared edible film has 23.8% scavenging activity compare the original one. The longer the reaction time and Kelakai extract concentration on edible film will gave higher scavenging activity; this is due to the high composition of Kelakai extract reacts as an antioxidant with DPPH.

4. Conclusions

The edible film-based cassava starch/eggshell composite containing antioxidant was successfully prepared by a simple method. The best addition of eggshell powder was 10%, wt/v in the presence of 10%, v/v Kelakai extract, produced cassava edible film with a thickness of 0.122 mm, tensile strength of 1.6 Mpa, elongation break of 25.4% and scavenging activity about 10.13% after 10 min.

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