



Development of antioxidant-rich edible film using eggshell and tapioca flours with kelakai (*Stenochlaena palustris*) extract

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ARTICLE INFO	ABSTRACT
<i>Article history:</i> Received: 17 April 2025 Received in revised form: 24 May 2025 Accepted: 25 May 2025	One of the major challenges in the food industry is the limited shelf life products. A promising approach to address this issue is the use of edible films—thin, consumable layers that protect food from environmental factors such as moisture, oxygen, and humidity. This study aimed to evaluate edible films based on varying ratios of chicken eggshell powder and tapioca flour, determine the optimum concentration of Kelakai (<i>Stenochlaena palustris</i>) extract as an antioxidant source, and assess the films' physical, chemical, and application properties. Edible films were prepared using eggshell powder (0-30% w/w), tapioca flour (5 g), distilled water (93.5 mL), Kelakai (<i>Stenochlaena palustris</i>) extract (0-10% v/v), and glycerol (1.5 g). The mixture was heated to $\pm 96^{\circ}\text{C}$ at 1000 rpm for 40 minutes, cast into 20×10 cm molds, and dried at 50°C for 24 hours. The best formulation—10% eggshell powder with 5% Kelakai (<i>Stenochlaena palustris</i>) extract—produced a film with 0.174 mm thickness, 1.5 MPa tensile strength, 27.4% elongation, and water vapor transmission rate of $10.0278 \text{ g/m}^2\cdot\text{h}$. FT-IR analysis revealed C-H, O-H, and C=O functional groups. Antioxidant activity showed 5.19% scavenging activity after 10 minutes. The film successfully preserved sausage for 7 days, demonstrating its potential to extend shelf life and serve as an eco-friendly packaging alternative. These findings highlight the potential of combining bioactive plant extracts and mineral-rich waste materials to develop functional edible films. The results open opportunities for the advancement of active packaging systems with antioxidant functionality and the integration of nanocomposite technologies to further enhance mechanical strength, barrier properties, and bioactivity. This eco-friendly approach contributes to sustainable packaging innovations, particularly in reducing reliance on synthetic plastics while improving food quality and safety.
<i>Keywords:</i> Antioxidant, edible film, Kelakai (<i>Stenochlaena palustris</i>) extract, chicken eggshell powder, tapioca flour	

1. Introduction

The global demand for sustainable and environmentally friendly packaging materials has led to significant research into biodegradable alternatives, particularly edible films made from natural polymers. One of the most promising bio-fillers for such applications is eggshell powder, which consists primarily of calcium carbonate and has been widely recognized for its potential in food fortification and various industrial applications [1]. Eggshell is abundant, renewable, and offers structural and functional properties beneficial for biodegradable film development [2].

Numerous studies have demonstrated the effectiveness of incorporating eggshell-derived materials into starch-based edible films. For instance, the addition of CaCl_2 extracted from eggshells to thermoplastic starch significantly improved the mechanical properties and extended the shelf life of food products [1]. Similarly, the integration of eggshell powder into cassava starch matrices has been shown to enhance film durability while maintaining biodegradability [3,4,5]. The mechanical reinforcement provided by eggshell fillers is attributed to their high calcium content and fine particle distribution, which can be optimized using analytical techniques such as X-ray diffraction (XRD), Fourier-transform

infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) [6,7,8,9].

In addition to their mechanical advantages, eggshell-based films can offer enhanced bioactivity. The incorporation of bioactive plant extracts, such as Kelakai (*Stenochlaena palustris*), has been explored for its antioxidant properties and potential to prolong food shelf life. Kelakai (*Stenochlaena palustris*) extract contains flavonoids and phenolics, making it suitable as a natural additive in smart packaging systems designed to reduce oxidative spoilage [10,11]. Edible films enriched with rosemary, red cabbage, rose, and butterfly pea flower extracts have been reported to exhibit similar antioxidative and functional characteristics [2,4,12,13] aligning with the principles of active and smart packaging technologies.

Several studies have also focused on combining eggshell with other biopolymers, such as gelatin, soy protein isolate, alginate, and polyvinyl alcohol (PVA), to develop composite films with improved barrier and tensile properties [9,14,15,16]. For example, soy protein-isolate and eggshell membrane conjugates loaded with eugenol have been developed into antimicrobial edible packaging [7], while ternary films combining eggshell, starch, and gelatin have shown enhanced structural integrity [17].

Moreover, the physico-mechanical and structural properties of these films have been extensively characterized. Techniques such as SEM provide insights into surface morphology and film uniformity [18], while FTIR and XRD are used to confirm

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molecular interactions and crystallinity in composite matrices [8,9,19]. These analytical tools are essential in optimizing film formulations and understanding the role of each component.

Beyond food packaging, eggshell has also been employed as a biodegradable heterogeneous catalyst, showcasing its versatility in green synthesis applications [20]. The growing interest in valorizing eggshell waste highlights the broader shift toward circular economy models and zero-waste strategies in material science.

In application, edible coatings incorporating functionalized eggshell fillers have been shown to effectively preserve perishable products like strawberries, supporting their role in extending shelf life through moisture barrier and antioxidant functionalities [21,22]. Recent innovations have also introduced alginate-based edible films reinforced with eggshell and nano-materials, offering further enhancements in bioactivity and mechanical strength [8,23]. Furthermore, recent advancements in edible film technology have focused on combining mineral-rich fillers with natural bioactive extracts to produce multifunctional packaging materials. For example, developed smart edible films based on alginate incorporated with butterfly pea flower extract and eggshell powder, demonstrating enhanced mechanical properties and antioxidant activity [24]. Meanwhile, eggshell waste can act as a multifunctional filler to overcome the limitations of starch-based films, improving both their structural integrity and bioactivity [25]. Altogether, the integration of eggshell powder and Kelakai (*Stenochlaena palustris*) extract into edible films represents a promising approach for developing biodegradable, functional packaging materials. This study aims to formulate and characterize edible films using varying compositions of eggshell powder and Kelakai (*Stenochlaena palustris*) extract, evaluate their physical, mechanical, and antioxidant properties, and assess their effectiveness in preserving sausage as a model food product. By focusing on the combination of bio-filler and plant-derived antioxidant, this research contributes to the development of active, sustainable packaging that aligns with nanocomposite and smart packaging technologies.

2. Materials and Methods

2.1. Materials

The materials used in this study included chicken eggshells collected from a local supplier in Banjarbaru, South Kalimantan; tapioca flour (Pak Tani brand) purchased from the Banjarbaru market; and Kelakai ferns (*Stenochlaena palustris*) sourced from Gambut, South Kalimantan. Additional chemicals used were glycerol ($C_3H_8O_3$), 1,1-diphenyl-2-picrylhydrazyl (DPPH), 95% methanol, silica gel, and distilled water.

2.2. Method

Young kelakai leaves were washed, oven-dried at 50 °C for 48 hours, blended into a fine powder, and sieved using a 40-mesh sieve. For extraction, 10 g of the powdered leaves was mixed with 100 mL of distilled water, heated at 50 °C for 60 minutes using a magnetic heated stirrer, then cooled and filtered. Chicken eggshells were similarly cleaned by washing, boiling for 15 minutes, and drying in an oven at 60 °C for 2 hours. The dried shells were blended into powder, sieved through a 40-mesh sieve, and stored in a desiccator until use. To prepare the edible film, a mixture of eggshell powder (0%, 5%, 10%, 15%, 20%,

30% w/w) and 5 g of tapioca flour was placed in a beaker, followed by the addition of Kelakai (*Stenochlaena palustris*) extract (0%, 2.5%, 5%, 7.5%, 10% v/v), 1.5 g of glycerol, and 93.5 mL of distilled water. The entire mixture was then heated to approximately 96 °C while continuously stirred at 1000 rpm for 40 minutes. Once homogenous, the solution was poured into a 20 × 10 cm mold and dried in an oven at 50 °C for 24 hours to form the edible film.

3. Results and Discussion

3.1. Characterization of edible film with the addition of flour chicken eggshell

The resulting chicken eggshell powder contains 98.81% calcium with impurities of 1.9% Co, S, Fe, Cu, Sr, Mo, Er, Lu components based on X-Ray Fluorescence (XRF) analysis. Previous research found the calcium content of chicken eggshells to be 95% and 5% organic compounds [3]. The eggshell used in this study contains more calcium, this difference is possible due to differences in the type of chicken eggs and the food consumed by the chicken.

The effect of chicken eggshell powder composition on edible film products with visualization observations can be seen in Figure 1.

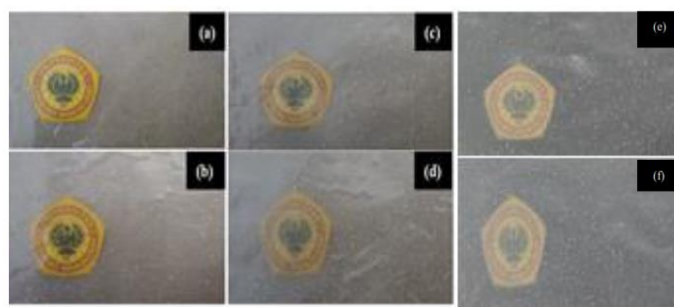


Fig. 1. Edible film products with variations of chicken egg shell flour composition (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%; (f) 30%.

The resulting edible film shows different brightness or translucency by placing objects under it. As the concentration of chicken eggshell powder increases, the level of brightness will decrease or opaque. Morphological observations by Scanning Electron Microscopy (SEM) are shown in Figure 2.

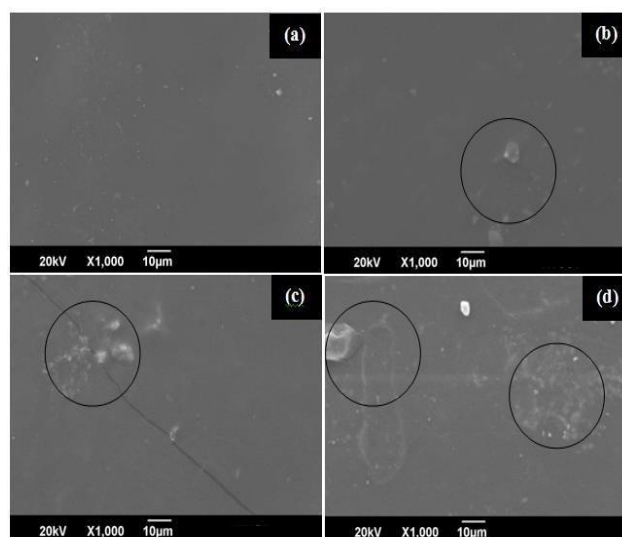


Fig. 2. SEM images of edible film with chicken egg shell flour concentration 0% (a), 10% (b), 20% (c), 30% (d) (w/w)

The sample without the addition of chicken eggshell powder as shown in Figure 2(a) exhibit edible film with smooth surface and evenly distributed while the edible containing 10% as demonstrated in Figure 3(b)-(d) shows some lumps (circle in the figure). The incorporation of eggshell powder led to a rougher surface and increased agglomeration with higher concentrations. This was evident in the SEM images, which showed uneven surfaces and the presence of clusters or lumps in the film matrix [5]. These lumps are chicken eggshell powder that fills part of the edible matrix. The greater the concentration of chicken eggshell powder, the more lumps on the surface of the edible film. The addition of chicken eggshell powder can affect the thickness of the edible, the results obtained are shown in Table 1. The higher the concentration of chicken eggshell powder, the thicker the edible film. This is due to the addition of chicken eggshell powder which is useful as a filler, if excessive, the constituent matrix will be tight and thickened [3]. The resulting thickness is still below the maximum edible film standard of 0.25 mm, so in terms of physical properties the resulting thickness meets the standards for edible films.

Table 1. Thickness of edible film with chicken egg shell addition

Shell Flour Concentration Chicken Egg (% w/v)	Thickness (mm)
0	0.174± 0.0114
5	0.176± 0.0148
10	0.184± 0.0195
15	0.180± 0.0212
20	0.194± 0.0245
30	0.200± 0.0335

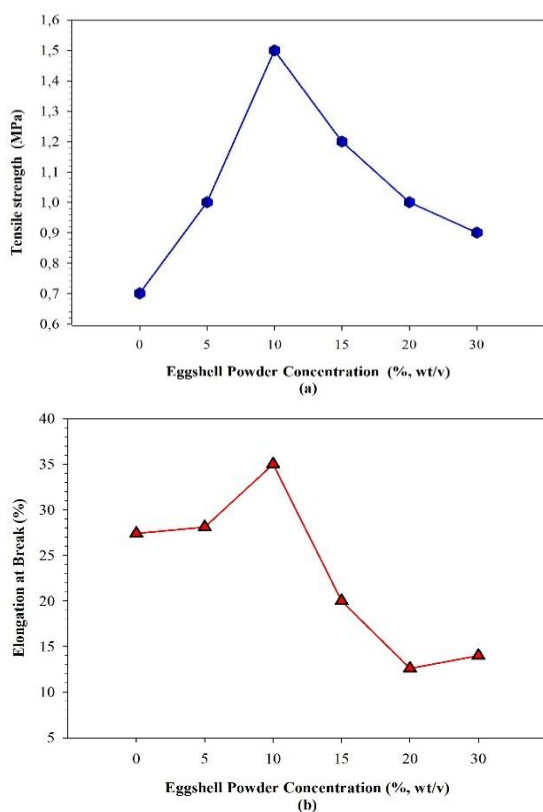


Fig. 3. Effect of chicken egg shell flour composition (0%, 5%, 10%, 15%, 20%, 30% (w/b)) on tensile strength test (a), and elongation at break of edible film (b)

Tensile strength test to determine the effect of chicken eggshell powder concentration on the tensile strength of edible film. Figure 3(a) shows the profile of tensile test values at various concentrations. The tensile strength of chicken eggshell powder composition samples 0%; 5%; 10%; 15%; 20%; 30% (w/b) were 0.7; 1; 1.5; 1.2; 1; 0.9 MPa respectively. Based on the results of the best edible film tensile strength test at 10% chicken eggshell powder composition where the highest tensile strength value is 1.5 MPa. The addition of eggshell powder as a matrix filler in the edible structure matrix provides an optimum value, if the concentration is excessive, the tensile strength will decrease because it affects the structural interconnection in the matrix. Analysis of the elongation at break test of edible film is shown in Figure 3(b). The addition of chicken eggshell powder gives a greater elongation at break effect, but at certain concentrations the value will decrease because too much filler material in the matrix results in reduced flexibility and elasticity of the edible film so that it breaks easily. This also applies to the tensile strength test which shows the same change profile. The concentration of adding 10% chicken eggshell powder gave the optimum value of elongation at breakage of 35%. An edible film developed using banana peel flour and chicken eggshell powder with the addition of cinnamon leaf extract (*Cinnamomum burmanii*) reported that incorporating eggshell powder improved the tensile strength and thickness of the edible film, while reducing water solubility, indicating a denser and more robust film structure [26]. These findings support the use of eggshell powder as a functional bio-filler that enhances the film's mechanical integrity and resistance to moisture. This aligns with our current findings, where eggshell powder contributed to improved mechanical characteristics of the film matrix.

Fourier Transformed Infra Red (FT-IR) analysis to analyze the functional groups contained in the resulting edible film can be seen in Figure 4.

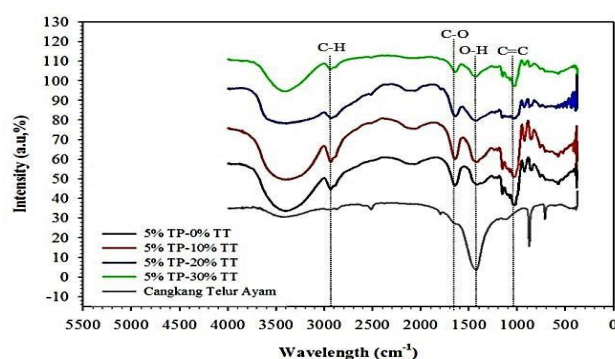


Fig. 4. FT-IR Analysis of various concentrations of chicken egg shell flour in edible film

Figure 4 shows the changes in functional groups in edible film due to the effect of the addition of chicken eggshell powder. The chemical structure of edible film is composed of four functional groups including C=C, C-H, O-H, and C=O groups. O-H group at 1459 cm⁻¹, C-H group at 2946 cm⁻¹, C=C group at 1083 cm⁻¹, and C=O group at wave number 1701 cm⁻¹. Eggshell powder has high absorbance of O-H, C=C, and C-H groups but the absorbance on edible film decreases with each composition. The composition of 10% eggshell powder in edible film with the highest adsorbance on O-H and C=O groups. One of the main characteristics of eggshells is the presence of a calcite structure similar to the mineral calcium carbonate (CaCO₃). The broad regions occur by overlapping spectra of hydrogen in the phosphate group, stretching of O-H structural vibrations and absorbed H₂O. The stretching intensity of C-O is observed in

broad and sharp regions. The medium broad region around C-H indicates the presence of CaCO_3 in eggshell powder.

Thus, the FT-IR spectrum of eggshell powder indicates the presence of calcite in the sample structure which further proves successful integration of eggshell powder into the film matrix [27] and is hygroscopic and has water moisture [20], contributing to the film's structural integrity [9].

3.2. Impact of Kelakai (*Stenochlaena palustris*) extract on edible film characteristics

The edible film containing 10% chicken eggshell powder was found to be the most optimal formulation. The effect of adding Kelakai (*Stenochlaena palustris*) extract on the edible film, based on visual observation, is shown in Figure 5.



Fig. 5. Edible Film Product 10% Chicken Egg Shell Flour Composition with Kelakai (*Stenochlaena palustris*) Extract Composition (a) 0%; (b) 2.5%; (c) 5%; (d) 7.5%; (e) 10% (v/v)

The addition of Kelakai (*Stenochlaena palustris*) extract serves as a source of antioxidants. As the concentration of the extract increases, the resulting edible film exhibits a more yellowish color, which is attributed to the natural brown hue of the extract. The incorporation of Kelakai (*Stenochlaena palustris*) extract is expected to enhance the functional properties of the edible film. Morphological observations using Scanning Electron Microscopy (SEM) are presented in Figure 6.

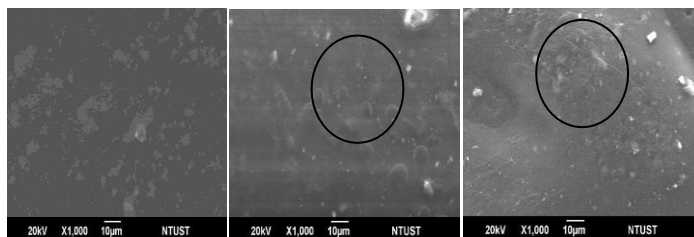


Fig. 6. SEM images of Kelakai (*Stenochlaena palustris*) extract Composition (a) 0%; (b) 5%; (c) 10%, (v/v) on Edible Film

SEM observations revealed that the addition of Kelakai (*Stenochlaena palustris*) extract fills the film matrix alongside the chicken eggshell powder. Figure 6 shows the formation of aggregates at each concentration level, with the size of the aggregates increasing as the extract concentration rises (indicated by circles in the image). The addition of Kelakai (*Stenochlaena palustris*) extract also affects the physical properties of the edible film, as shown in Table 2.

As the concentration of the extract increases, the resulting edible film becomes thinner and more difficult to form. This is likely due to the antioxidant compounds increasing the

hydrophobicity of the film surface, caused by a small amount of carnosic acid migrating to the surface during the drying process [13]. The tensile strength test of the edible film showed no significant changes, indicating that the addition of Kelakai (*Stenochlaena palustris*) extract does not affect its tensile strength. However, the elongation at break slightly decreased due to the formation of voids within the film matrix, as seen in Figures 6(b) and 6(c), marked by the presence of aggregates or bubbles in the film. This results in reduced elasticity of the edible film. FT-IR analysis was conducted to identify the functional groups present in the produced edible film. The FT-IR spectrum is shown in Figure 7.

Table 2. Physical Properties of Edible Film with the Addition of Kelakai (*Stenochlaena palustris*) extract

Concentration Kelakai (<i>Stenochlaena palustris</i>) extract (% v/v)	Thickness (mm)	Tensile Strength (MPa)	Elongation Termination (%)
0	0,184 ± 0,0167	1,5	35,0
2,5	0,178 ± 0,0840	1,5	30,0
5,0	0,174 ± 0,0114	1,5	27,4
7,5	0,132 ± 0,0130	1,6	25,8
10,0	0,122 ± 0,0084	1,6	25,4

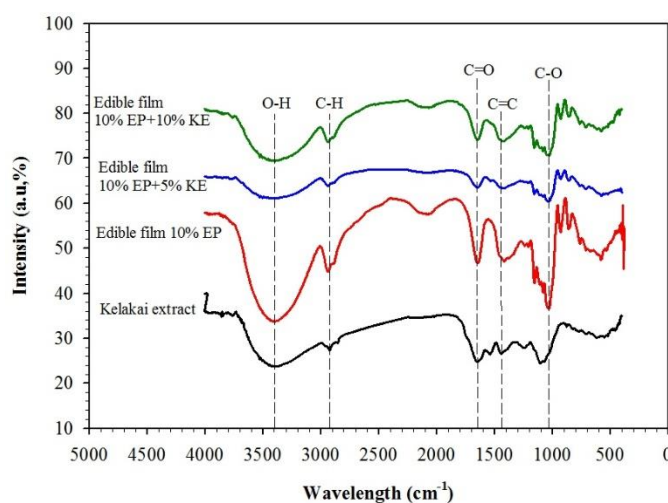


Fig. 7. FT-IR Analysis of Various Concentrations of Kelakai (*Stenochlaena palustris*) extract on Edible Film

Figure 7 shows the changes in functional groups of the edible film influenced by varying concentrations of Kelakai (*Stenochlaena palustris*) extract (0%, 5%, and 10% v/v). The chemical structure of the edible film consists of several functional groups, including O-H, C-H, C=O, C=C, and C-O. The O-H group appears around 3400 cm^{-1} , C-H at 2927 cm^{-1} , C=O at 1714 cm^{-1} , C=C at 1497 cm^{-1} , and C-O at 1027 cm^{-1} . The Kelakai (*Stenochlaena palustris*) extract exhibits strong absorbance for O-H, C=O, and C-H groups; however, the absorbance intensity in the edible film decreases as the extract concentration increases, indicating an interaction and successful incorporation [9] between the extract and the film matrix. Edible films containing rosemary extract have shown a greater reduction in O-H groups compared to those without extract, which may be attributed to interactions between the extract and

the glycerol–starch matrix, altering the availability of O–H within the polymer network. Films incorporating extracts also display signals corresponding to aromatic ring stretching, due to the phenolic compounds in the extract, appearing near 1515 cm^{-1} . Another notable peak around 1714 cm^{-1} is associated with the C=O group from carboxylic acids [13].

3.3. Analysis of water vapor transmission, antioxidant activity, and practical application of the edible film

The effect of Kelakai (*Stenochlaena palustris*) extract composition (0%, 2.5%, 5%, 7.5%, and 10% v/v) on Water Vapor Transmission Rate (a) and Antioxidant Activity (b) of the edible film is presented in Figure 8.

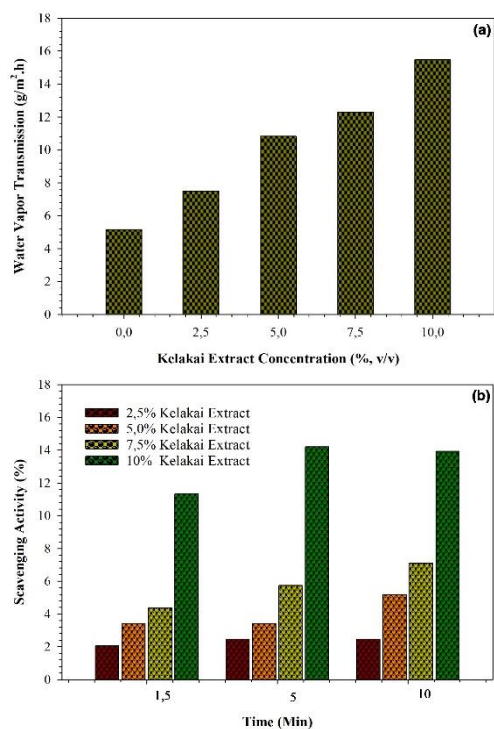


Fig. 8. Effect of Kelakai (*Stenochlaena palustris*) extract Composition 0%, 2.5%, 5%, 7.5%, 10% on (a) Water Vapor Transmission Analysis, and (b) Antioxidant Activity of Edible Film

Figure 8(a) shows that increasing the concentration of Kelakai (*Stenochlaena palustris*) extract leads to a higher water vapor transmission rate. The transmission values for Kelakai (*Stenochlaena palustris*) extract concentrations of 0%, 2.5%, 5%, 7.5%, and 10% (v/v) were 5.1597, 7.8931, 10.0278, 12.3056, and 15.4792 g/m²·h, respectively. This increase is attributed to the extract causing a less compact matrix, making the edible film more permeable to water vapor. Antioxidant compounds can enhance the hydrophobicity of the film surface, likely due to the migration of small amounts of carnosic acid during the drying process [13].

Figure 8(b) presents the antioxidant activity analyzed using DPPH (1,1-diphenyl-2-picrylhydrazyl) solution to evaluate radical scavenging efficiency, indicated by the reduction of the purple color to colorless over a 10-minute observation. The scavenging activity values for Kelakai (*Stenochlaena palustris*) extract concentrations of 2.5%, 5%, 7.5%, and 10% (v/v) were 2.4590%, 5.1913%, 7.1038%, and 13.9344%, respectively. In comparison, pure Kelakai (*Stenochlaena palustris*) extract exhibited an activity of 58.61%. The longer the reaction time between the edible film and DPPH, the greater the free radical scavenging activity observed, due to the higher content of

Kelakai (*Stenochlaena palustris*) extract acting as an antioxidant. The research demonstrated that the addition of Kelakai (*Stenochlaena palustris*) extract enhanced the antioxidant activity of the edible films. This was attributed to the presence of phenolic compounds in Kelakai, which are known for their antioxidant properties [10].

Antioxidant activity analysis of Kelakai (*Stenochlaena palustris*) extract (KE) after 10 minutes and its application on sausage wrapping are shown in Figure 9.

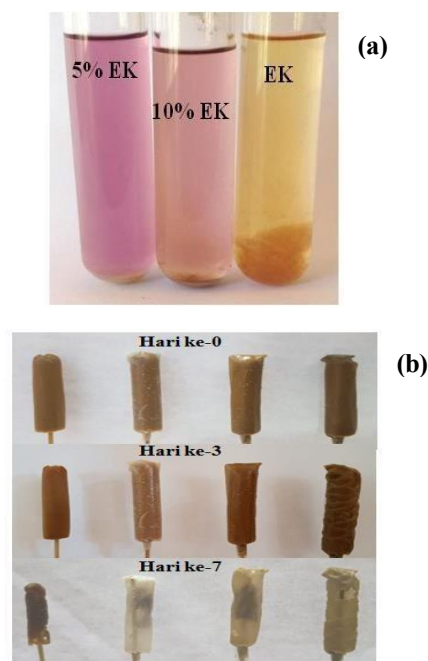


Fig. 9. Antioxidant Activity Analysis of Kelakai (*Stenochlaena palustris*) extract (KE) after 10 minutes (a), and Observation of Edible Film Application on Sausage (b)

The color change observed after 10 minutes for each concentration of Kelakai (*Stenochlaena palustris*) extract showed varying degrees of purple discoloration removal. The pure Kelakai (*Stenochlaena palustris*) extract demonstrated the highest antioxidant activity, as indicated by the most significant color change. The application test of the edible film was conducted by wrapping food—sausages were chosen as the test sample—and observing changes and the time taken for spoilage. Spoilage was determined by the appearance of microorganisms on the food, with daily observations made under open-air conditions. The edible film application test included four variations: unwrapped sausage, sausage wrapped with edible film without extract, sausage wrapped with edible film containing 5% extract, and sausage wrapped with 10% Kelakai (*Stenochlaena palustris*) extract. On the third day, all sausages were still in relatively good condition. However, the sausage wrapped in edible film with 10% extract appeared sticky due to higher moisture content, which promoted interaction between water molecules and the sausage surface. By the seventh day, all sausages showed signs of shrinkage caused by exposure to the open air. The 10% extract film exhibited noticeable degradation. It can be concluded that edible film with 5% Kelakai (*Stenochlaena palustris*) extract effectively preserved the sausage for up to 7 days. Similarly, rosemary-based edible films have been reported to provide food protection for up to 7 days [13]. The effects of adding Belimbing Wuluh (*Averrhoa bilimbi*) leaf extract, a natural antioxidant, to a carrageenan–chitosan-based edible film demonstrated a significant enhancement in the antioxidant activity of the film without compromising its

mechanical properties [28]. For further development, adding chitosan to corn starch-based edible films improved the tensile strength and significantly reduced the water vapor transmission rate (WVTR), which are crucial parameters for food packaging applications [29]. Their findings highlight the synergistic effect of biopolymer and additive combinations in enhancing barrier properties to create functional packaging. These results parallel our observations where the incorporation of eggshell powder and Kelakai (*Stenochlaena palustris*) extract contributed to decreased WVTR and elevated antioxidant activity while maintaining acceptable film structure, thus demonstrating potential for practical application as protective food packaging.

4. Conclusion

The optimal composition of chicken eggshell powder in combination with tapioca flour for producing edible film was found to be 10%. This formulation resulted in favorable physical characteristics, including a thickness of 0.184 mm, tensile strength of 1.5 MPa, and elongation at break of 35%. Additionally, the most effective concentration of Kelakai (*Stenochlaena palustris*) extract was determined to be 5%, which produced films with a thickness of 0.174 mm, tensile strength of 1.5 MPa, and elongation at break of 27.4%. At this concentration, the water vapor transmission rate was 10.0278 g/m²·h, while the antioxidant activity reached 5.1913%. The edible film incorporating 5% Kelakai (*Stenochlaena palustris*) extract also demonstrated good performance when applied to sausage preservation, maintaining its protective properties for up to seven days. These results suggest that the combination of eggshell powder and Kelakai (*Stenochlaena palustris*) extract in a tapioca-based edible film not only improves the mechanical, barrier, and antioxidant properties of the film, but also enhances its potential for use as an eco-friendly, functional packaging material. This edible film offers promising applications in food preservation and sustainable packaging technologies. Further development could explore its incorporation with other bioactive compounds or its scalability in commercial food packaging industries.

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