Shelf-life prediction of pineapple dodol packed with edible film using accelerated shelf life tests

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Abstract. The accelerated shelf-life tests (ASLT) can be applied to predict the shelf life of products to reduce time and cost. ‘Dodol’, semi-wet food, is easily damaged by microbial activity. This study aims to predict the shelf life of pineapple dodol packed by an edible film with and without the addition of antioxidant and antimicrobial. ASLT with the Arrhenius model was used to estimate the shelf life of the dodol products. The dodols with different packaging were stored at some temperatures of 20, 30 and 40 °C for 42 days, analyzed periodically to determine total plate count, moisture content, and free fatty acid. The results showed that the shelf life of dodol packed with polypropylene plastic was the longest. In addition, dodol wrapped with edible film with antimicrobial-antioxidant had a longer shelf life than that of without antimicrobial-antioxidant. Based on the total plate count, the shelf life of dodol wrapped with polypropylene plastic, the edible film with antimicrobial-antioxidant, and the edible film without antimicrobial-antioxidant were 32, 26, and 22 days, respectively.

1. Introduction

Dodol is one of the traditional foods which is quite popular in Malaysia and Indonesia [1,2]. Dodol is a type of semi-wet food that is potentially damaged due to microbial growth and the hydrolysis process. Pineapple dodol is generally prepared from glutinous rice flour, sugar, extract of ripe pineapple and sometimes with the addition of permitted food additives [3,4].

Currently, consumers demand high food quality that will be maintained at a high level during the period of purchase and consumption. Pineapple dodol decreased in the moisture content and increased in free fatty acid and total plate count during storage at room temperature [4]. The shelf life of dodol is one of the important attributes that should be informed to consumers. Food industries need a relatively short time to determine the shelf life of their products. They usually use accelerated test techniques that considerably shorten the process of obtaining the data, compared to the actual storage time which needs a long time [5]. Accelerated test stores product under a range of environmental conditions (generally relative humidity or temperature) and provides kinetic data. The kinetic data shows the deterioration process of the food quality as a function of time-based on reaction change [6]. The Arrhenius model can be applied to know the rate of a chemical reaction to the changes in temperature which the differences of temperature can vary from 5 to 10°C [7].

Packaging has an important role to extend the shelf life of dodol. Usually, dodol is wrapped with plastic-based packaging. Unfortunately, plastic is non-edible, non-biodegradable and not environment friendly. Nowadays, there has been an increasing interest in the use of edible materials to pack foods [8]. The alternative packaging that is biodegradable to wrap dodol is an edible film [2–4,9]. The edible
film has been studied as a potential packaging for seaweed [9] and pineapple dodol [3,4]. Accelerated Shelf Life Testing (ASLT) method has been studied to predict the shelf life of seaweed dodol [10], sugar palm fruit jam [11], and gudeg [12]. However, there are still limited studies about the ASLT of dodol packed by an edible film that incorporated with antioxidant and antimicrobial. Therefore, the aim of this study is to predict the shelf life of pineapple dodol packed with edible film with the addition of antioxidant and antimicrobial. This study can provide information about the opportunities for using eco-friendly food packaging and estimating its shelf life.

2. Materials and Methods

2.1. Materials
The materials used in this study was arrowroot starch obtained from ‘Kusuka Ubiku’ (Yogyakarta, Indonesia), carrageen and cocoa butter purchased from Setia Guna (Bogor, Indonesia), glycerol, beeswax, tween 80, ascorbic acid, and sodium benzoate obtained from Brataco (Bandung, Indonesia), and pineapple dodol produced by SME Alam Sari (Subang, Indonesia).

2.2. Research procedure
The edible film was made based on a procedure reported by Ratnawati [13] with a composition of arrowroot starch 1%, carrageen 1%, glycerol 1%, tween 80 0.2%, sodium benzoate 0.03%, beeswax, cocoa butter, and ascorbic acid each of 0.1% and the remaining aquadest. Starch and carrageen were dissolved separately in distilled water until the solution became clear. Solutions were then mixed and added with glycerol, tween 80, melting beeswax and cocoa butter, then stirred until homogeneous. Then, the edible solution was degassed under a vacuum for 1 min and casted on acrylic plates (20 x 20 cm). Dry at 50°C for 24 h before applying as packaging. This film was an edible film without the addition of antioxidant-antimicrobial (CF), while an edible film with addition antioxidant-antimicrobial (AAF) was prepared by decreasing the temperature of the control edible solution to 70°C and added ascorbic acid and sodium benzoate.

18 g Pineapple dodol was wrapped using packaging according to treatment. The treatment in this study was the packaging type to wrap dodol, namely (1) edible film without the addition of antioxidant-antimicrobial (CF), (2) edible film with the addition of antioxidant-antimicrobial (AAF), and (3) polypropylene packaging (PP). Dodol with PP treatment was packed with polypropylene plastic and put individually in a ‘mica’ plastic (polyvinylchloride type plastic) as secondary packaging as many as 6 pieces. Dodol with CF and AAF treatment was wrapped with edible film and put in 0.001mm thick PP Plastic every 6 pieces to be sealed and put in mica plastic. Dodol samples were then stored in incubators at 20, 30 and 40°C. Dodol was analyzed on water content, free fatty acid (FFA), and total plate count (TPC) every week (seven days for six weeks).

2.3. Product analysis
The moisture content, free fatty acid, and total plate count of the sample were determined following to Indonesian national standard (SNI 01-2986-2013). The gravimetric method was used to measure the moisture content of the sample. About 2 g of dodol were weighed into pre-dried weighing bottles and dried at 105 °C in an electric oven (Memmert Instrument) till achieved constant weight.

The free fatty acid of dodol was measured by extracting dodol for 2 hours with n-hexane using a Soxhlet. The extracted fat was heated over the water bath until all the solvent evaporated and left behind the fat residue. Then, it was dissolved with 50 ml neutralized alcohol and titrated with sodium hydroxide solution of 0.1M using PP indicator. FFA content was calculated according to equation (1)

\[
FFA = \frac{V \times N \times 20}{W} \times 100
\]  

(1)

where FFA is a free fatty acid (%), V is a volume of NaOH solutions (ml), N is the normality of NaOH solutions (N), and W is sample weight (g).
Total plate count (TPC) of dodol was measured by weighing about 5 g of samples placed aseptically in a sterile Erlenmeyer. Each sample was added with 45 ml BPW (Buffer Peptone Water) and homogenised until dilution required. Pipe 1 ml of each dilution to petri-dish, then poured 15-20 ml Plate Count Agar (PCA) media at 45°C. After the media solidified, the petri-dish was incubated for 48 hours at 35-37°C. Total plate count was determined according to equation (2)

$$TPC = n \times F$$  \hspace{1cm} (2)

where TPC is total plate count (colony per gram), n is the average of the colony from two petri dishes from one dilution (colony per gram) and F is dilution factors of the average colony.

2.4. Determination of shelf life

The shelf-life of dodol is determined by the Accelerated Shelf Life Test (ASLT) method using the Arrhenius equation. The kinetic equation used to estimate shelf life may be expressed as:

$$dt = k[A]^{n}$$  \hspace{1cm} (3)

it is time, k is the kinetic constant, [A] is the change in concentration of a tested component, and n is order of reaction. The steps to determine the reaction order (n) are as follows [14]:

1. guess reaction order;
2. integrate; linearize; plot experimental data in the graph of the relationship between storage duration and the data (if data fit a straight line, then the guess is right, if not start again);
3. choose the slope (reaction rate constant) that gives the highest coefficient of determination (R²); plot k value (slope of graph) against the experimental temperature according to the Arrhenius equation below (equation 4-5) [7].

$$k = k_0 \exp(-E_a/RT)$$  \hspace{1cm} (4)

$$\ln k = \ln k_0 - (E_a/R) \times (1/T)$$  \hspace{1cm} (5)

Where $k_0$ is the rate constant, $E_a$ is the activation energy, R is the gas constant (1.9872 cal/mol°K), and T is the absolute temperature (°K). The slope of plot between ln $k$ versus 1/T is $E_a/R$.

3. Results

3.1. Moisture content

Information about the moisture content of food is very important because this parameter relates to the change of physical and chemical properties of the product. The moisture content of dodol packed in various types of packaging during storage at different temperatures can be seen in Table 1.

| Packaging type | Days | 0  | 7  | 14 | 21  | 28  | 35  | 42  |
|----------------|------|----|----|----|-----|-----|-----|-----|
| T = 293 K PP  |      | 14.28 | 13.45 | 13.43 | 12.57 | 12.94 | 12.77 | 12.71 |
| T = 293 K CF  |      | 14.28 | 12.73 | 11.92 | 12.99 | 11.59 | 11.89 | 9.61 |
| T = 293 K AAF |      | 14.28 | 12.35 | 12.13 | 11.24 | 11.64 | 12.04 | 10.72 |
| T = 303 K PP  |      | 14.28 | 12.01 | 11.69 | 11.88 | 11.39 | 10.68 | 11.01 |
| T = 303 K CF  |      | 14.28 | 12.19 | 11.09 | 12.20 | 12.26 | 9.87 | 10.41 |
| T = 303 K AAF |      | 14.28 | 11.27 | 12.75 | 12.43 | 11.49 | 11.03 | 10.49 |
| T = 313 K PP  |      | 14.28 | 11.05 | 10.35 | 11.57 | 10.21 | 9.52 | 9.09 |
| T = 313 K CF  |      | 14.28 | 10.23 | 10.49 | 9.79 | 9.69 | 8.60 | 9.40 |
| T = 313 K AAF |      | 14.28 | 11.83 | 11.78 | 11.12 | 10.26 | 9.07 | 9.10 |

The moisture content of dodol met SNI 01-4296-1996 which requires a maximum moisture content of 20%. Generally, the moisture content of dodol that was packed in all types of packaging decreased during the storing period. It is possibly due to the evaporation of water vapor from dodol system to the
environment system. The same result was reported by Indrianti [4], the moisture content of dodol that was packed with plastic PP and edible film decreased to 9.72% and 9.82%, respectively after stored at ambient temperature for five weeks. In addition, Warkoyo [9] exhibited the weight loss of dodol packed with the edible film due to loss of water content and other volatile compounds during storage. The different result was reported by [2], there was an increase in water content during storage. Plastics PP had the ability to prevent water loss higher than edible films AAF and CF because its water vapor transmission rate is lower than edible films [13].

3.2. Free fatty acid
The free fatty acid values of pineapple dodol packed with plastic and edible film were illustrated in Figure 1. The initial FFA of dodol was 0.0198% then increased to range 0.0587-0.0877% after stored for six weeks at various temperatures. These values had fulfilled the Indonesian national standard of dodol (SNI 2986-2013) which required FFA below 0.5%. Free fatty acid (FFA) is formed due to hydrolysis of triacylglycerol and decomposition of hydperoxides in fat at system temperatures in the presence of air and moisture [15]. The increase in FFA content is due to the oxidation reaction of fats contained in dodol by oxygen in the environment. Indrianti [4] showed a similar result that the FFA content of pineapple dodol packed in plastic PP and heat-treated edible film increased after 5 weeks of storage by 0.069%-0.075%.

![Figure 1. Free fatty acid of pineapple dodol during storage in various packaging types and temperatures.](image)

3.3. Total plate count
The microbes number (TPC) in pineapple dodol packed in plastic and edible film when stored in various temperatures is presented in Figure 2. The initial TPC of dodol was 116 colony/g which had met the Indonesian national standard of pineapple dodol (SNI 01-4296-1996) that is below 500 colony/g. After four weeks stored in various temperatures showed an increase in TPC value above the allowed standard. Similar results were reported by [2,4] that during storage there was an increase in total plate count. The final TPC of pineapple dodol packed in plastic PP and the heat-treated edible film was 1400 colony/g and 1500 colony/g after 5 weeks of storage. The addition of sodium benzoate in edible film AAF resulted in a similar ability with plastic PP to prevent microbial growth, while dodol which was wrapped with an edible film without antimicrobial (CF) gave the highest TPC value. The increased temperature resulted in an increased TPC value. The effect of storage temperature on microbial growth is explained by the Arrhenius relationship that the microbial growth rate increases with increasing temperature till the optimum temperature range comes.
3.4. Basic reaction kinetics to estimate the decreasing quality of dodol

The linearization of equation (3) using data in Table (1), Figure (2), and Figure (3) produce linear regression equations with each value of the coefficient of determination ($R^2$) is shown in Table 2. The choice of the reaction order is done by comparing the $R^2$ value in each linear regression equation at the same temperature of the zero-order reaction (A plotted against time) and first-order reaction (ln A plotted against time). The chosen reaction order is a reaction order with a larger value of $R^2$. Table 2 shows the values of $R^2$ first order were greater than that one of $R^2$ zero-order especially at 303°C and 313°C for moisture content and free fatty acid parameter, meanwhile for TPC parameter zero-order had $R^2$ value greater than first order.

| Packaging type                      | Moisture content parameter | FFA parameter | TPC parameter |
|-------------------------------------|----------------------------|---------------|---------------|
|                                     | Order 0 | Order 1 | Order 0 | Order 1 | Order 0 | Order 1 | Order 0 | Order 1 |
| Poly propylene plastic (PP)         |          |          |          |          |          |          |          |          |
| T = 293K                            | 0.709    | 0.702    | 0.934    | 0.983    | 0.934    | 0.948    |          |          |
| T = 303K                            | 0.706    | 0.724    | 0.890    | 0.897    | 0.930    | 0.908    |          |          |
| T = 313K                            | 0.704    | 0.735    | 0.888    | 0.897    | 0.866    | 0.863    |          |          |
| Edible film without antimicrobial- antioxidant (CF) |          |          |          |          |          |          |          |          |
| T = 293K                            | 0.734    | 0.716    | 0.804    | 0.777    | 0.953    | 0.935    |          |          |
| T = 303K                            | 0.729    | 0.744    | 0.951    | 0.948    | 0.993    | 0.989    |          |          |
| T = 313K                            | 0.617    | 0.644    | 0.951    | 0.964    | 0.975    | 0.956    |          |          |
| Edible film with antimicrobial- antioxidant (AAF) |          |          |          |          |          |          |          |          |
| T = 293K                            | 0.648    | 0.651    | 0.866    | 0.861    | 0.964    | 0.931    |          |          |
| T = 303K                            | 0.617    | 0.617    | 0.857    | 0.878    | 0.917    | 0.895    |          |          |
| T = 313K                            | 0.913    | 0.933    | 0.905    | 0.921    | 0.879    | 0.868    |          |          |

The slope values of each linear regression equations, which give a greater value of $R^2$, can be seen in Table 3. This slope value is the kinetic constant. All packaging types exhibited the same pattern that with the increasing of temperature, the kinetic constant (k) also rose. The driving force to evaporate water in food is larger when the environment temperature rises. The rise of FFA kinetic constant by increasing storage temperatures is due to a high temperature transfer rate of oxygen to food system enlargement [16] which promotes the fat oxidation process. The nutrient content of the food can affect...
microbial growth. Besides water, in order to grow, microorganisms require a carbon source for energy and the easiest way is by decomposing carbohydrates like sugar contained in dodol. The decomposition rate of carbohydrates also increases at high temperature to support microbial growth (Steele, 2004). Sarungallo [16] reported the same results for free fatty acid content of red fruit oil, as well as Anggraini [11] for moisture content of sugar palm fruit jam.

Table 3. The slope (reaction rate constant) of linear regression equation which give the highest $R^2$.

| Packaging type               | Parameter | MC   | FFA  | TPC  |
|------------------------------|-----------|------|------|------|
| Polypropylene (PP)           | $T = 293K$ | 0.002 | 0.0289 | 0.0180 |
|                              | $T = 303K$ | 0.005 | 0.0322 | 0.0196 |
|                              | $T = 313K$ | 0.008 | 0.0345 | 0.0224 |
| Control film (CF)            | $T = 293K$ | 0.006 | 0.0281 | 0.0235 |
|                              | $T = 303K$ | 0.007 | 0.0317 | 0.0309 |
|                              | $T = 313K$ | 0.008 | 0.0328 | 0.0313 |
| Antimicrobial-antioxidant film (AAF) | $T = 293K$ | 0.004 | 0.0274 | 0.0205 |
|                              | $T = 303K$ | 0.005 | 0.0290 | 0.0252 |
|                              | $T = 313K$ | 0.010 | 0.0349 | 0.0280 |

Plotting on the graphic above kinetic constant value versus $1/T$ (K$^{-1}$) resulted in linear regression equations according to equation (5). The constant value of these equations can be seen in Table 4. According to equation (5), the slope value of the equation is the value of $-E/R$ from the Arrhenius equation, so that it can be obtained the activation energy of dodol on each packaging type. The kinetic constant ($k$) was calculated following equation (2).

Table 4. Shelf life of dodol on some packaging types.

| Parameter | MC, % | FFA, % | TPC, colony/g |
|-----------|-------|--------|---------------|
|           | PP    | CF     | AAF           | PP   | CF  | AAF |
| Linear equation constant : | | | |
| ln (ko)   | 15.61 | -0.61  | 8.65          | 0.76 | -1.12 | 0.14 | -0.61 | 0.82 | 1.02 |
| $-E/R$    | 6377  | 1319   | 4176          | 814  | 713  | 1103 | 1000 | 1327 | 1434 |
| $R^2$     | 0.97  | 0.99   | 0.91          | 0.99 | 0.92 | 0.90 | 0.98 | 0.80 | 0.97 |
| E, cal/mol| 12671 | 8298   | 8298          | 1617 | 1417 | 2191 | 1987 | 2637 | 2850 |
| k         | 0.004 | 0.007  | 0.006         | 0.032| 0.035| 0.034| 0.020| 0.028| 0.024|
| Shelf life, days | 66 | 44 | 49 | 197 | 180 | 183 | 32 | 22 | 26 |

The kinetic constant ($k$) indicates the deterioration rate of food. The greater the deterioration rate, the faster the product is damaged, so then the shelf life of the product was getting shorter. Table 4 shows for all quality parameters of dodol, polypropylene plastic (PP) had the lowest ‘k’ value followed by an edible film with the addition of antioxidant-antimicrobial (AAF) and edible film control without antimicrobial-antioxidant addition (CF). Eom [17] proved that covering rice cake with thicker surface coating led low water vapor permeability and strong water holding capacity. The thickness of plastic PP, edible film AAF, and edible film CF was 0.042 mm, 0.061 mm, 0.059 mm, respectively, while their water vapor permeability was 1.22, 24.94, 23.62 g/m²/24hours [13,18]. It promotes preventing moisture loss so that the shelf life of dodol packed with plastic PP is the longest compared to the other packaging types. Ascorbic acid contained in the edible film acts as an antioxidant, which can effectively capture free radicals and cut off radical reactions, thereby inhibiting the increase in FFA. It implied to lengthen
the shelf life of *dodol* packed with edible film AAF. As well, antimicrobial (sodium benzoate) added in the edible film can inhibit microbial growth, thus the shelf life of *dodol* packed with edible film AAF is longer than that of packed with edible film (CF). The edible film made from heat-moisture treated sweet potato starch had a similar ability with plastic PP to prevent water loss and to inhibit the rate of increase in FFA content during storage [4].

Based on Equation (3) the shelf life of food product is determined by the kinetic constant (k) and the concentration gradient of a tested component (A). One of the parameters that affect the ‘k’ value is activation energy (E) which indicates the energy needed to change the quality attribute. The greater the activation energy value, the greater energy needed to react, so that the changes in quality occur more slowly. It was supported by the activation energy (E) value of total plate count which was lower than that of moisture content. Gradient concentration to calculate shelf life is the difference between critical quality and initial quality. The critical value of tested attributes was determined based on the Indonesian national standard of *dodol*. Even though the FFA attribute had activation energy smaller than TPC, but it showed a greater gradient concentration, so that the shelf life based on the FFA parameter was longer than that of the TPC parameter. This indicated that *dodol* was very easily damaged due to microbial activity.

**4. Conclusions**

The shelf life of pineapple *dodol* was predicted using Accelerated Shelf Life with the Arrhenius approach with a number of parameters, namely moisture content, total plate count, and free fatty acid. Polypropylene plastic showed the best inhibition to prevent deterioration of food quality. The presence of antimicrobial-antioxidant in the edible film could decrease the quality deterioration rate of *dodol*. Total plate count was a critical parameter which determined the shelf life of *dodol*. The predicted shelf-life of *dodol* packed with polypropylene plastic, an edible film with antimicrobial-antioxidant, and edible film without antimicrobial-antioxidant at 30°C were 32, 26, and 22 days, respectively.

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