Shelf life prediction of carica seeds powder using accelerated method

S D Astuti*, S Lestari, Erminawati, S Widarni, G Wijanarko and F N Wibawa
Jenderal Soedirman University. Dr. Soeparno Street, Karangwangkal Purwokerto
Jawa Tengah Indonesia 53122

Corresponding author: santi.astuti@unsdo.ac.id

Abstract. Carica fruit is a main commodity in Dieng plateau. Its seeds is one of the by-products in carica fruit cocktail processing. Carica seeds contain high antioxidants, so that it can be consumed as a functional beverage. The objectives of this study was to determine the shelf life of the product. The processes involved in carica seed powder production were: soaking with citric acid, boiling, fermentation with *R. oligosporus* inoculum, drying, roasting, and mixing the roasted seeds with dried jackfruit at ratio of 3:1. The packaging used in this study were glass jar and aluminum foil. The products were stored at 35, 45, and 55°C and analyzed for its chemical and sensory characteristics at every 5 d for 20 d. The shelf life was examined by the acceleration method with Arrhenius equation using moisture content as the critical parameter. Results of the study showed that shelf life of products packed with aluminum foil at room temperature (25°C) was 1 year and 1 month and at cold temperature (8°C) was 1 year 4 months. Meanwhile, the shelf life of products packed in glass bottles at room temperature (25°C) was 5 months and at cold temperatures (8°C) was 6 months.

1. Introduction
Carica is a main commodity and indigenous fruit from Dieng plateau. The carica fruit appearance was similar to papaya fruit but the size is smaller, the taste is sour, and the aroma was stronger than papaya. Carica fruit rich in Vitamin C, potassium, flavonoid, antioxidant, and dietary fiber. It can only be consumed after being processed. One of the products is carica cocktails which is made from unripe fruit. The use of over-ripe fruit and by product of cocktails production (pulp, seed) have not been carried out. Carica seeds rich in antioxidant but the flavor is bitter and astringent. Like papaya seeds, carica seeds contain antioxidants, such as terpenoids, saponins, and alkaloids [1,2]. The solid state fermentation process using commercial inoculums containing *R. oligosporus* in 0.2% of concentration and 75h of time fermentation could reduce the bitter taste of carica seeds. The addition of dried jackfruit in carica seeds can improve the sensory quality with ratio of 28%:72% respectively. This product can be consumed as functional beverage as it contains antioxidant, phenol, vitamin C, sugar, kalium, and pottasium, i.e 89.21%db, 0.35%db, 135.25mg/100g db, 10.94%db, 90.65ppm, and 51.66ppm, respectively. Beverage made from carica seeds powder produce beverage has actual rating score (range = 1–9) of overall acceptability, fruity flavor, bitter taste, and after taste i.e 8.23, 7.76, 4.41, 3.79, respectively [3]. Products were packaged with several package alternatives to extend shelf life and facilitate transportation and distribution, such as glass jar and aluminum foil. Both are inert
and have a low water vapor transmission rate. Glass jar has small pores and thick [4]. Aluminum foil is thin, lighter and more flexible [5].

Product shelf life is the time interval between production and consumption where the product still has good quality based on its nutritional, sensory properties, and safety [6,7]. Shelf life needs to be determined to ensure product quality and safety for consumers [8]. The accelerated shelf life testing with Arrhenius model can predict the product shelf life quickly [9]. This method has been widely used to examine product shelf life, such as instant coffee [10]. The concept used is Q10 where every 10°C increase in temperature, the rate of deterioration will be faster. This principle has been used to determine shelf life of salacca fruit [11]. The quality testing during storage was carried out by medium shelf life principle, where products were tested every 5 days for 20 days [12]. Product was stored at a temperature higher than room temperature to accelerate its deterioration [13]. The shelf life was determined using water content as critical parameter because the product was hygroscopic which means it easily absorbs water from the environment and to form agglomerates when stored. This properties was similar to instant coffee [10]. In addition, from previous research, it was proven that water content produced the highest $R^2$ value compared to other variables.

The objective of this research was to predict the shelf life of carica seed powder stored in glass jar and laminated aluminum foil pouch using the acceleration method and the Arrhenius model.

2. Experimental details

2.1. Ingredients
Carica fruit was obtained from Wonosobo district and jack fruit from Purwokerto district. Other ingredients (sugar, commercial inoculums, glass jar, and laminated aluminum foil pouch) were obtained from a local market in Purwokerto central Java.

2.2. Methods
The stages of this research were: 1) preparing materials, tools and other instruments; 2) making products with the optimum formula (results from previous research); 3) packing the product in glass jar and aluminum foil; 4) storing the product in an incubator with a temperature of 35, 45, and 55°C; 5) testing water content of product at storage for 5, 10, 15, and 20 days by Gravimetric method [14]; 6) analyze and process data to determine the reaction order kinetics and Arrhenius equation; 7) determine the shelf life prediction of carica seeds powder.

2.2.1. The production of Carica seed powder. Carica seeds were obtained from the waste of cocktails production by SMEs in Wonosobo. Carica seeds were separated from fruit flesh by washing in running water. A total of 3 kg Carica seeds were soaked with 6L of a citric acid solution of 0.20% w/v for 1h and then soaked with 6 L of water for 8h. After draining, Carica seeds were steamed for 1h. After cool down, Carica seeds were mixed with 0.2% w/v commercial inoculum (La Prima), packed in perforated plastic (dimensions of length x width x height = 20 cm × 20 cm × 0.5 cm), and then fermented for 72h. Fermented Carica seeds were steamed for 10 min, dried with a cabinet dryer (60°C of temperature) for 8h, roasted using low heat for 45min up to 1h. Dried Carica seeds were ground with a seed grinder and sifted with a 60 mesh sieve to produce Carica seed powder [3], with slightly modification.

2.2.2. The production of dried jack fruit. Jackfruits with medium maturity were processed to separate the flesh and seeds. After washed, fruits were steamed for 4 minutes over medium heat. After cooled down, the fruits were dried with a cabinet dryer (60°C of temperature). Dried jackfruits were ground with a fruit grinder to produce jackfruit powders [3], with slightly modification.

2.2.3. The production of carica seed beverages. The main ingredients consisted of Carica seed powders and dried jackfruits in a ratio = 3 : 1. Carica seed beverage was made by brewing 5% of the
main ingredient with 5% brown sugar using boiling water. After the mixture was left for 5 min, the mixture was filtered to separate the liquid and solid parts [3], with slightly modification.

2.2.4. **Shelf life prediction test using arrhenius model.** The water content of carica seed powder was plotted against time and three equations for different product storage temperatures, \( Y = bx + a \) will obtained, where “\( x \)” is the storage time (day), “\( Y \)” represents the characteristic value of carica seed powder, “\( a \)” is the initial characteristic value of carica seed powder, and “\( b \)” is the rate of characteristic change (slope). The quality decrease rate (\( k \)) obtained from every linear regression equation. Then, the \( \ln k \) value was plotted with \( 1/T \) (K-1), and the intercept and slope value of the linear regression equation, \( \ln k = \ln k_0 - (E / R) (1 / T) \) was obtained. The Arrhenius equation was calculated after activation of energy characteristic of carica seed powder and constant value “\( k_0 \)” was obtained. It was calculated by this formula: \( k = k_0 \cdot e^{-E/RT} \) (1), where: \( k \) = quality degradation constant, \( k_0 \) = constant (independent of temperature), \( E \) = activation energy, \( T \) = absolute temperature (K), \( R \) = gas constant (1.986 cal / mol K). The rate of reaction (\( k \)) from the changes characteristic of carica seed powder at a pre-determined temperature (\( T \)) can be calculated by Arrhenius equation. The determination of shelf life of carica seed powder was done by measuring water content as critical parameter because it has the highest correlation coefficient (\( R^2 \)) compared to other parameter. Furthermore, “\( k \)” value obtained was entered into order equation of reaction: \( At = Ao + kt \) (2), where: \( Ao \) = A value at the beginning of shelf life, \( At \) = A value at the end of shelf life; \( t \) = shelf life; \( k \) = quality degradation constant. From this stage, the prediction of shelf life of carica seed powder for each specified temperature will be obtained [13].

3. **Results and discussion**

3.1. **The profile of water content**

Carica seed powder has water absorbing properties, like coffee and milk powder. Therefore, the critical parameter used to determine shelf life of product was water content. This research was in line with the shelf life study of instant galangal coffee [15], full cream milk powder [16], and instant coffee [10]. In this study, the critical water content value used was 17.98%, according to the shelf life study with the same method for instant coffee [10].

The results showed that the water content increased with the length of storage time and the higher in storage temperature (Figure 1). The increase in the water content during storage for products packed in glass jar was higher (4.51% wb) than those in laminated aluminum (4.06%). Aluminum foil packaging has better water protection properties than glass jar packaging. At high temperatures, the permeability of the packaging increase, especially in glass jar, so that the water vapor transmission rate also increases. This condition causes an increase in water content during the storage of products in glass jar is higher than with aluminum foil. In addition, the type of aluminum foil used to package carica seed powder was laminated by plastic. The results of this research is in line with a storage studies on tomato powder [17]. Product storage for 20 days causes an increase in water content from 3.88%wb to 4.64%wb. The appearance of carica seed powder is particles with a small diameter so that it has a large surface area. This condition causes product absorb water easily from the environment, which is indicated by the agglomeration of the product during storage. The same results was obtained in the storage of Alpinia galanga powder [15]. The increasing of storage temperature from 35°C to 55°C caused an increase in water content from 4.38%wb to 4.41%wb. The increase in temperature causes an increase in the permeability of the packaging, thereby accelerating the absorption of water from the environment especially in products that has high of hygroscopic properties such as carica seed powder.
3.2. The determination of the reaction order kinetics

The determination of kinetic order reaction is a way to predict quality degradation. The zero order reaction indicates a constant rate of damage. It was detected by plotting water content as y-axis and storage time as the x-axis. The first order reaction indicates the logarithmic or exponential rate of the damage. It was detected by plotting the ln value of water content as the y-axis and storage time as the x-axis. The choice of kinetic order reaction was based on the linear regression equation from zero-order and first order reaction.

Table 1. Slope, intercept, and $R^2$ value of the zero and first order reaction

| Packaging      | Temperature (°C) | Zero order | First order |
|----------------|------------------|------------|-------------|
|                |                  | Slope (k)  | Intercept   | R²           | Slope (k)  | Intercept   | R²           |
| Aluminium foil | 35               | 0.017      | 3.821       | 0.82        | 0.004      | 1.342       | 0.83         |
|                | 45               | 0.023      | 3.903       | 0.90        | 0.005      | 1.362       | 0.91         |
|                | 55               | 0.024      | 3.911       | 0.86        | 0.006      | 1.364       | 0.87         |
| Glass jar      | 35               | 0.051      | 3.885       | 0.97        | 0.012      | 1.361       | 0.98         |
|                | 45               | 0.059      | 3.931       | 0.91        | 0.013      | 1.373       | 0.92         |
|                | 55               | 0.075      | 3.925       | 0.87        | 0.016      | 1.373       | 0.88         |

The kinetic order reaction used is the one with high $R^2$ value [18]. The higher value of the determination coefficient show higher accuracy of data. The results showed that the average value of $R^2$ of products stored in glass jar and aluminum foil from the first order reaction was higher than zero order. This shows that the increase on water content of carica seed powder was exponential. The slope value states the relationship between water content and storage time. The results showed a positive slope value, which means that decrease in product quality was indicated by an increase on water content. The increase on water content was due to the hygroscopic properties of product [17]. The slope, intercept, and $R^2$ values of the zero and one order reactions of the carica seed powder stored in glass jar and aluminum foil packaging can be seen in Table 1 and Figure 2.

3.3. The determination of arrhenius equation and “K” value

Based on the Arrhenius approach, the changes in product quality were strongly influenced by temperature. The Arrhenius equation is the relationship between the value of ln (natural log) k from the first order regression equation and the value 1/T in °K units. The Arrhenius equation was obtained by plotting the value of ln k as the “y” axis and 1 / T as the “x” axis. The resulting Arrhenius equation
is a constant value of deterioration (K). The results of the Arrhenius equation water content at 3 storage temperatures were presented in Table 2. The graph of the correlation between ln k and 1/T of the product with aluminum foil and glass jar packaging can be seen in Figure 3 and Figure 4.

**Table 2.** The Arrhenius Equation of carica seed powder pack in different packaging

| Kemasan       | T (˚C) | K   | ln k | T (˚K) | 1/T (˚K) | Pers. linier ln K vs 1/T | Pers. Arrhenius ln K = ln K₀ – Ea/R |
|---------------|--------|-----|------|--------|----------|--------------------------|------------------------------------|
| Alumunium foil| 35     | 0.004 | -5.47| 308    | 0.0032   | y = 0.18-1728.6x         | ln K = 0.1761+1728.6x               |
|               | 45     | 0.005 | -5.18| 318    | 0.0031   | R² = 0.87                 |                                    |
|               | 55     | 0.006 | -5.13| 328    | 0.0030   | y = 1.09-1712.5x         | ln K = 1.806+1712.5x               |
| Botol kaca    | 35     | 0.012 | -4.46| 308    | 0.0032   | R² = 0.97                 |                                    |
|               | 45     | 0.013 | -4.34| 318    | 0.0031   |                            |                                    |
|               | 55     | 0.016 | -4.11| 328    | 0.0030   |                            |                                    |

**Figure 2.** The correlation curve between ln k and 1 / T of the product with aluminum packaging

From Table 2 and Figure 2, it can be seen that the Arrhenius equation for carica seed powder in aluminum foil is "ln K = 0.18 + 1728.6 (1 / T)". At a temperature of 35˚C, the "ln k" value is -5.47 or the "k" value is 0.004. It shows that the increase in water content of products packed in aluminum foil was 0.004 units per day. At a storage temperature of 45˚C, the increase in water content was 0.005 units per day, and at a storage temperature of 55˚C, the increase in moisture content was 0.006 units per day. The Arrhenius equation for carica seed powder in a glass jar is "ln K = 1.81 + 1712.5 (1 / T)" (Figure 3). At a temperature of 35˚C, the "ln k" value was -4.46 or the "k" value was 0.012 which indicates that the increase in water content of products packaged in glass containers is 0.012 units per day. At a storage temperature of 45˚C, the increase in water content was 0.013 units per day. At a temperature of 55˚C, the increase in water content was 0.016 units per day.
3.4. The determination of shelf life prediction

The equation obtained from the correlation curve is in line with the Arrhenius equation. The value of the deterioration constant (K) of the Arrhenius equation is obtained from calculating the slope of the regression equation between the “ln” water content and the analysis time at different temperatures. Based on the equation "y = 0.1761-1728.6x" and "y = 1.09 -1712.5x", the aluminum foil "ln k0" value is 0.18, the glass bottle "ln k0" value is 1.81. The "Ea / R" value of aluminum foil is 1712.5, and the "Ea / R" value of the glass jar is 1728.6. The Arrhenius constant is calculated by substituting the temperature value using the equation "ln K = ln k0 - Ea / RT". Shelf life is calculated from the difference between the initial water content value and the predetermined critical water content. The prediction of shelf life (ts) of the product is calculated using the equation “ts = (ln No – ln Nt) / KT” (Table 3).

![Figure 3. The correlation curve between lnk and 1 / T of the product with glass jar packaging](image)

![Figure 4. The curve of the relationship between storage temperature and shelf life of carica seed powder](image)

The shelf life of packed carica seed powder in aluminum foil packaging at storage temperatures of 35, 45, and 55°C, i.e. 349, 293, and 248 days, respectively; whereas in the glass jar were 133, 112, and 95 days, respectively. From Figure 4, it can be seen that the shelf life of products packed with aluminum foil was longer than glass jar. The water vapor transmission rate of glass jar packaging was higher than aluminum foil so that protection against water penetration into the material of aluminum foil packaging is better than glass jar, especially in extreme storage conditions such as high temperatures [10,17]. Food products are usually stored at room temperature (about 25°C) or cold temperature in the refrigerator (about 8°C). The prediction of shelf life of carica seed powder stored at
room temperature with aluminum foil packaging is 397 days (1 year 4 months), while glass jar is 151 days (5 months). The prediction of shelf life of carica seed powder stored at cold temperatures with aluminum foil packaging is 483 days (1 year 6 months), while glass jar is 183 days (6 months).

**Table 3.** Arrhenius constant “K” value dan shelf life prediction of carica seed powder with variations of packaging and storage temperature

| Packaging      | Temperature (˚C) | ln K   | K     | Shelf life (days) |
|----------------|------------------|--------|-------|-------------------|
| Aluminium foil | 35               | -5.436 | 0.004 | 349               |
|                | 45               | -5.259 | 0.005 | 293               |
|                | 55               | -5.094 | 0.006 | 248               |
| Glass jar      | 35               | -4.474 | 0.011 | 133               |
|                | 45               | -4.299 | 0.014 | 112               |
|                | 55               | -4.135 | 0.016 | 95                |

4. Conclusion

Laminated aluminum packaging provides better protection against water penetration into the product than glass jar, mainly in high of temperature storage. The increase of temperature and storage time lead to the increasing of water content. The decrease in the quality of carica seed powder during storage is indicated by an increase in water content. The predicted shelf life of carica seed powder that packed in aluminum foil at room temperature (25°C) is 1 year and 1 month and at cold temperature (8°C) is 1 year 4 months; meanwhile, products packed in glass jar at room temperature (25°C) is 5 months and at cold temperatures (8°C) is 6 months. The recommendations for packaging and storage conditions with consideration of efficiency and economics for carica seed powder is aluminum foil package at room temperature.

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