The effect of the use of perkamen paper packaging on the shelf life estimation of Bolu Cukke using the Accelerated Shelf Life Testing (ASLT) method

R Latief and E Safitry
Universitas Hasanuddin, Makassar, 90245 South Sulawesi, Indonesia
E-mail: rindamias04@yahoo.com

Abstract. This study aims to estimate the shelf life of Bolu Cukke packaged in parchment paper using the Accelerated Shelf Life Testing (ASLT) method. The critical water content parameters in Bolu Cukke are its aroma and its texture. The equilibrium moisture content was obtained from six types of saturated salt solution in the humidity chamber with 7-84% RH. Isothermal sorption curves are obtained by plotting Aw with equilibrium moisture content (Me) which would be tested using five isothermal sorption equation designs to obtain the Mean Relative Deviation (MRD) value. The lowest MRD value is 9.2 obtained from Hasley’s Isothermal equation design. All variables were then collected using the Labuza equation. The results of this study indicated that the estimation of shelf life of Bolu Cukke products in the parchment paper packaging at 30°C with 78% RH is approximately 7 days.

1. Introduction
In South Sulawesi, there are special characteristics of Bolu Cukke, especially in the Soppeng regency. Bolu Cukke is a well-known Buginese cake with a sweet taste, easily found in the community, especially in the Buginesse (Soppeng) community. Bolu Cukke is one of the Buginesse food widely produced in several areas of Sulawesi, including Soppeng.

Bolu Cukke is made of wheat flour and rice flour as the main ingredients. In addition to the wheat flour and rice flour, Bolu Cukke is added with sugar and palm sugar, which gives an appetizing aroma and color to the cake. Due to its appetizing taste and soft texture, this cake is widely preferred. Bolu Cukke is also known as Bolu Cungkil. Since Bolu Cukke is a semi-moist cake, it is undoubtedly perishable. Bolu Cukke has a short shelf life.

To ensure that food products are safe to consume and free of any damages, information about the product’s shelf life is highly required. The shelf life of food products is a period of products that is sensory and nutritionally acceptable and safe for consumption, especially for perishable food products[1].

To determine the shelf life of Bolu Cukke, the Accelerated Shelf Life Testing (ASLT) method is used. The reason of using this method is the use of parameter with environmental conditions in the test process which is able to accelerate the product degradation. The advantage of this method is its relatively short testing time with exact precision and accuracy [2]. It is expected that by knowing the shelf life or expiration time of Bolu Cukke, the safety of Bolu Cukke to consume is guaranteed.
In this method, the packaging condition of Bolu Cukke is set outside the normal conditions by using paper. Paper packaging is chosen since it is categorized as a product that is environmentally friendly, easy to process, recyclable, biodegradable, and relatively cheaper.

The type of paper used in this research is parchment paper. It is used for its resistance to grease and its good wet strength even in boiling water. Its surface is not fibrous, odorless, tasteless, and transparent, making the paper often called glacin paper. Also, it does not have good inhibition to gas unless it is coated with certain materials [3].

Based on the description above, this research was conducted with the title “The Effect of the use of Parchment Paper Packaging on the Shelf Life Estimation of Bolu Cukke Using the Accelerated Shelf Life Testing (ASLT) Method”.

2. Methodology

2.1. Measurement of initial moisture (Mi)
The empty cup was dried in an oven at the temperature of 105°C set for an hour. Then, it was cooled in a desiccator for 15 minutes. After that, it was weighed (W1). A total of 5 grams of sample (W2) was put in a cup and then stored in the oven at the temperature of 105°C for three hours until it reached a constant weight. The cup containing the sample was cooled in a desiccator and then weighed (W3). The initial moisture content was calculated using the following formula:

2.2. Measurement of critical moisture content (moisture critical, Mc)
Samples were stored at 76% of RH using saturated NaCl solution. Periodically (every three days), a panelist acceptance test for the product aroma was carried out. The average acceptance test score was calculated daily. When the average value reached 2 (dislike), it was determined that the product was in a critical condition. The critical water content was measured using the oven method, as shown in point 1. Then, the critical water content was calculated using the following formula:

\[ KAMc = \frac{(W_2 - W_3 - W_4)}{(W_3 - W_1)} \times gH_2O/gsolid \]

2.3. Determination of isothermal sorption curves
The saturated salt solution was prepared. The amount of salt was weighed and put in the humidity chambers. The required amount of salt and water as shown in table 1.

| The saturated salt solution | RH (%) | Quantity |
|-----------------------------|--------|----------|
|                             |        | Salt (gram) | Water (mL) |
| NaOH (H₂O)                  | 7      | 150       | 8          |
| MgCl₂·6H₂O                  | 32     | 200       | 25         |
| K₂CO₃                       | 43     | 200       | 90         |
| KI                           | 69     | 200       | 50         |
| NaCl                         | 76     | 200       | 60         |
| KCl                          | 84     | 200       | 80         |

Source: Agus (2004)[4]

It was stirred, and a certain amount of water was added until it was saturated in order to maintain solution saturation in order that the relative humidity resulted is unchanged, and the absorption remains in progress. Humidity chambers were closed and left for 24 hours at the temperature 30°C. 5 grams of the packaged Bolu Cukke were taken. Bolu Cukke was hanged in a humidity chamber containing a saturated salt solution. Samples were weighed periodically (every 24 hours) to reach the
constant weight, indicating that the equilibrium water content has been reached. The moisture of the samples with constant weight was measured using the oven method and expressed on a dry basis as shown in point 3.3.1. Isothermal sorption curves were made by plotting water content and balance water activity. Water activity (a_w) was calculated by dividing the RH value of each humidity chamber by 100.

2.4. The determination of isothermal sorption design
The value of Moisture Equilibrium (Moisture Equilibrium, Me) and a_w was included in the sorption equations of Chen Clayton, Henderson, Hasley, Caurie, and Oswin. The five isothermal sorption equation designs are evaluated for the Mean Relative Deviation (MRD) value. If the MRD value is <5, it means the isothermal sorption design can describe the actual situation precisely. If 5 <MRD is <10, the design is rather accurate to describe the real situation, and if MDR is 10, the design does not accurately describe the real situation.

\[
MRD = \frac{100}{n} \sum_{i=1}^{n} \left| \frac{Mi - Mpi}{Mi} \right|
\]

Mi = Tested Moisture content
Mpi = Calculated moisture content
N = Number of data

2.5. The determination of supporting parameters
The packaging permeability value \( \left( \frac{k}{x} \right) \) is obtained from literature references. The value of saturated vapor pressure (Po) at the temperature 30°C is obtained from the Labuza table. The value of b (slope of the curve) is obtained from the gradient of the selected isothermal sorption equation design. The value of the cross-sectional area (A) is obtained by multiplying the package dimensions. The total solids value (Ws) is obtained by correcting the total weight of the sample subtracted with the initial moisture content.

2.6. Shelf life estimation
All the measured and applied parameters in the previous step, including Mi, Mc, Me, k/x, Po, b, A, and WA, were integrated in the Labuza equation.

\[
\Theta = \ln \left( \frac{Me-Mi}{Me-Mc} \right) \frac{k}{x} \left( \frac{A}{Ws} \right) \frac{Po}{b}
\]

Information:
\( \Theta \) = Estimated shelf lifetime (day)
Me = Product’s equilibrium moisture content (g H_2O/g solid)
Mi = Product’s initial moisture content (g H_2O/g solid)
B = Slope of isothermal sorption curve
Mc = Critical moisture content (g H_2O/g solid)
k/x = Water vapor permeability (g/m^2.day.mmHg)
A = Packaging surface area (m^2)
Ws = Product’s dry weight in the packaging (g solid)
Po = Saturated vapor pressure (mmHg)
3. Results and discussion

3.1. Proximate analysis of bolu cukke

The initial quality characteristic of Bolu Cukke observed in this study includes the chemical and organoleptic analysis. The parameter of chemical analysis observed is moisture content, fat content, protein content, and carbohydrate content. The result of the initial quality characteristics of Bolu Cukke can be seen in Table 2.

| Parameter           | Composition (%) |
|---------------------|-----------------|
| Moisture Content    | 0.29            |
| Ash Content         | 1.7             |
| Fat Content         | 3.64            |
| Protein Content     | 9.63            |
| Carbohydrate Content| 56.03           |

Source: Health Laboratory of Makassar, 2019

The result of the initial quality characteristics of Bolu Cukke above shows that Bolu Cukke has 0.29% of moisture content, 1.7% of ash content, 9.63% of protein content, and 56.03% of carbohydrate content.

3.2. Parameters of bolu cukke’s shelf life estimation

The shelf life of Bolu Cukke is determined by the Accelerated Shelf Life Testing method with a critical moisture approach. The main principle of this approach is determining the equilibrium moisture content (Me) of Bolu Cukke stored in various RH. Furthermore, the relation between the equilibrium moisture content of Bolu Cukke and storage RH will produce an isothermal curve. This curve can be used to find out the water vapor pattern of Bolu Cukke in the environment, and therefore the shelf life of ice cream can be determined using the Labuza equation.

The shelf life of the product calculated with the Labuza equation is shelf life at 78% RH. This RH is chosen to represent the storage condition of Bolu Cukke by consumers. Meanwhile, the required parameters to determine the shelf life estimation of Bolu Cukke using the Accelerated Shelf Life Testing method are initial moisture content (Mi), critical moisture content (Mc), equilibrium moisture content (Me), isothermal sorption curve determination, isothermal sorption design determination, the slope of isothermal sorption curve (b) determination and other supporting parameters determination such as packaging permeability (\( \frac{\theta}{2} \)), solids weight per packaging (Ws), packaging surface area (A), and saturated vapor pressure in the storage room (Po).

3.2.1. Initial moisture content (Mi).

Initial moisture content is the percentage of early water content possessed by foodstuffs and calculated immediately after being produced. Determined initial moisture content using Moisture Analyzer. The initial moisture content of Bolu Cukke is determined at the beginning of storage. Bolu Cukke is a semi-moist food that has a moderate moisture content percentage. According to the research result, the initial moisture content of Bolu Cukke is 29.23% DBT. Defines semi-moist food as the food with moisture content that is not too high and not too low between 15%-55% of wet basis and between 0.65-0.85 of Aw (water activity)[10].

3.2.2. Moisture content (Mc).

Critical moisture content is the amount of water content possessed by a product that is no longer acceptable to consumers organoleptically. It is determined using the Moisture Analyzer tool equipped with analytical balance and aluminum disc to facilitate the determination of sample weight with a heat temperature of 110°C, in which the moisture content in Bolu Cukke will be absorbed completely in 15 minutes that can produce output data in %. Graining is a critical parameter
that determines the damage of Bolu Cukke. It is a condition in which sugar crystals rise to the surface and make Bolu Cukke look dull and even moldy due to water absorption on the surface of the product. The critical moisture content of Bolu Cukke is determined when it is no longer acceptable to consumers organoleptically.

In this study, Bolu Cukke was stored at a room temperature of ±30°C at 76% of RH using sodium chloride. Every three days, the acceptance rate of the product was assessed by 15 panelists with the score range 1-5. Score 2 (dislike) is considered as the consumers’ acceptance threshold for Bolu Cukke. It began to receive a rejection on day 9. The graphic of the relationship between panelists’ acceptance rate and storage time can be seen in figure 1.

![Organoleptic](image)

**Figure 1.** Diagram of the relationship between acceptance test score bolu cukke and storage time.

Figure 1 shows that the average score of the highest acceptance test is found at the beginning of storage (day 0) with a score 4.7. The more days, the more the average acceptance test score decreases. On the 9th day of storage, the average acceptance test score reaches 2.1, which indicates that Bolu Cukke has reached its critical condition based on its appearance. After the determination of its critical condition, the next step is to calculate the moisture content. Bolu Cukke has a critical moisture content of 0.2028 g H₂O/g solid or 20.28% DBT. From the comparison with the initial moisture content, it can be concluded that Bolu Cukke has experienced a decrease in moisture content. This indicates that there has been evaporation of moisture content in Bolu Cukke to its surrounding environment, nearly reaching the balance, and thus the weight of moisture content will decrease. The relative humidity of air is lower than the relative humidity of materials; therefore, the materials will release water into their surrounding environment [5].

3.2.3. Equilibrium moisture content (Me). Equilibrium moisture content (Me) is used to describe the isothermal sorption curve of a product. Equilibrium moisture content is the amount of water content of materials after being in an environmental condition for a long period of time [5]. The equilibrium moisture content for this study was obtained by conditioning Bolu Cukke in six types of saturated solutions, where each of them formed a different RH. A saturated salt solution was made by dissolving the amount of salt until it was saturated. Meanwhile, the six types of salts used included NaOH, MgCl₂, K₂CO₃, KI, NaCl, and KCl. The temperature of 30 ±1°C of the six types of saturated salt solutions has RH consecutively 7%, 32%, 43%, 69%, 76%, and 84%. The various selected relative humidity value in this study aims to represent the water activity range as a whole and to obtain the most flawless and precise isothermal sorption curve to determine the product’s shelf life [4].

During the storage in various RH, there will be an interaction between the product and its environment. The vapor will transmigrate from the environment to the product or vice versa until it
reaches a balanced condition. The condition in which the product no longer absorbs or releases vapor to the environment is obtained. Similar to the determination of initial moisture content and critical moisture content, the determination of equilibrium moisture content also uses Moisture Analyzer. The equilibrium moisture content of foodstuffs will be obtained as indicated by the food weight’s constancy. The food weight is considered constant if the weight difference about three consecutive times weighing is not more than 2 mg/g at ≤90% RH and not more than 10 mg/g at >90% RH[6].

During the storage, the samples stored at 7, 32, 43, 69, 76, and 84 RH experienced a tendency to a reduction of food weight. This reduction happened because Bolu Cukke will experience a desorption process (weight reduction) to obtain equilibrium conditions with its environment. The equilibrium moisture content and the time to reach a balanced condition in this study can be seen in the table below. The stored samples will experience a weight increase or decrease, which shows a hydration characteristic phenomenon. Hydration characteristics of foodstuffs can be implied as physical characteristics, which include the interaction between foodstuffs and water molecules in the surrounding air. Meanwhile, the processes that happen during the sample storage at various RH’s are the absorption process and desorption process. Absorption is the process of moisture absorption by materials from the environment. Meanwhile, desorption is the process of moisture release of materials to the environment [7].

3.2.4. Isothermal sorption curve. Isothermal sorption curve is a curve depicting the relation between the moisture content in foodstuffs and the water activity (aw) or the relative humidity (ERH) of the storage room[7]. The isothermal sorption curve of Bolu Cukke can be seen in Figure 2.

![Figure 2. Isothermal curve of bolu cukke.](image)

In this study, the sorption isotherm curve was made by plotting the equilibrium moisture content value of the research results in Table 03 with the water activity value (aw). Figure 2 shows that the isothermic sorption curve of Bolu Cukke product is similar to the isothermic sorption curve type II and is sigmoidal, although it is imperfect. Each food product has a unique isothermic curve but is generally sigmoid. This sigmoid shape is due to the fact that food ingredients generally consist of a mixture of several components. Each food ingredient has a typical isothermic sorption curve. This depends on the moisture absorption pattern of each product [7].

3.2.5. Isothermic sorption equation design. Isothermic sorption curve equation design obtained from the research results with existing designs was carried out to obtain high curve smoothness. Many mathematical equation designs have been developed to explain the isothermic sorption phenomenon theoretically. However, in this study, only five mathematical equation designs were selected, such as Chen Clayton, Henderson, Hasley, Caurie and Oswin. These equation designs were chosen because they were able to describe isothermic sorption curves over a wide range of water activity
values. Moreover, these designs only had two parameters, so they were easy to use. If the purpose of using the isothermic sorption curve is to obtain high curve smoothness, it is better to choose simpler equation designs that have fewer parameters [8].

In order to simplify the calculations, the mathematical equation designs used were transformed from non-linear equations to linear equations so that the constant values could be determined using the least-squares design. The linear equation of the Bolu Cukke product from the isothermic sorption curve equation designs can be seen in table 3.

### Table 3. Equation of isothermic sorption curve of bolu cukkeproduct and Its Mean Relative Deviation (MRD) value.

| Design       | Equation                                              | Nilai MRD |
|--------------|-------------------------------------------------------|-----------|
| Chen Clayton | ln [ln(1/aw)]=5.24-25.97 Me                           | 15.7      |
| Henderson    | log [ln(1/(1-aw)]=-3.685+5.945 log Me                 | 18.24     |
| Hasley       | log [ln(1/aw)]=3.801-5.398 log Me                    | 9.02      |
| Caurie       | ln Me= -1.823+0.599 ln [aw/(1-aw)]                    | 11.38     |
| Oswin        | ln Me=-1.524+0.0110 aw                                | 14.71     |

Table 3 shows the MRD value of each equation of the isothermic sorption design. The highest curve smoothness of the five designs of the equation can be elevated through a design evaluation by calculating the value of Mean Relative Deviation. It is obvious that these designs can draw curves precisely, somewhat precisely, and imprecisely. Based on the MRD value obtained, the Hasley equation design is used as a reference in making the next isothermic curve, although the Caurie equation design has a value of 11.38. Theoretically, this value describes the isothermic curve imprecisely because its MRD value is larger than 10, so it is with Henderson, Oswin, and Chen Clayton design that equally describes imprecise isothermal conditions. The isothermal sorption design, which can describe the real situation very accurately is a design that has an MRD value of below 5%[11]. However, the MRD value of the Hasley equation design is lower, which indicates that the Hasley design is more coincident with the actual equilibrium conditions (reliable). The smaller the MRD value, the more precise the design is in describing the phenomenon of occurred isothermic sorption[11].

Chen Clayton, Henderson, Caurie, and Oswin equation designs whose values are 15.7, 18.24, 11.38, and 14.71 of each describe the whole isothermic curve imprecisely (MDR > 10). The suitability of each isothermic design to the isothermic of food products depends on the range of aw and the type of constituent of the food products [2]. The comparison of research isothermic sorption curve with Hasley equation design isothermic curve can be seen in figure 3.
3.2.6. Slope value (b) of isothermic sorption curve. The calculation of shelf life based on the Labuza equation requires slope (b) of the isothermic sorption curve. The value of the slope of the Bolu Cukke can be seen in figure 4.

The slope (b) of the isothermal sorption curve is determined from the straight line formed on the curve of the isothermic sorption equation design formed in the Hasley design [7]. Based on Figure 4, it is known that the connection points between water activity and equilibrium moisture content have a linear equation $y = a+bx$. The b value of the linear equation is the slope of the isothermic sorption curve. The value of slope (b) of the isothermic sorption curve in the Bolu Cukke product is 0.137.

3.2.7. Supporting parameters for estimating shelf life. Bottled water vapor permeability ($k$), packaging surface area (A), solid weight (Ws), and pure vapor pressure in the storage (Po) are supporting parameters in estimating the shelf life of the Bolu Cukke product. It is necessary to know the water vapor permeability value of the packaging type to estimate the shelf life of the product, which is calculated using the Labuza equation.

Each type of packaging has different water vapor permeability. Bottled water vapor permeability is the rate of water vapor transmission through one unit area with a certain thickness due to differences in water vapor pressure between the product and its environment [6]. The packaging used can affect
the quality of the packaged food, i.e., the occurrence of physical and chemical changes due to the migration of chemical substances from the packaging material to food and the changes in aroma, color, and texture caused by the transfer of water vapor and oxygen. The relationship between the type of packaging material and the durability of the packaged food material is determined based on its permeability. Permeability is the transfer of water or gas molecules through packaging, either from the product to the environment or vice versa. Water vapor permeability of the package is the speed of water vapor transmission through a unit area of material with a certain thickness due to differences in water vapor pressure between the product and the environment at a certain temperature and humidity [6]. The type of packaging used to package the Bolu Cukke product is propylene plastic. The type of plastic has a permeability of 0.0739 gH\textsubscript{2}O/m\textsuperscript{2}.day.mmHg[8]. Propylene type plastic is a good type of plastic as a barrier to water vapor since it has a fairly low water vapor permeability. The smaller the value of water vapor permeability of packaging is, the longer the shelf life of packaged food products is [9].

The size of the packaging surface area also affects the shelf life of a product. Area determination of the packaging is done by multiplying the length and width of packaging used. The wider the surface area of the packaging used, the higher the water vapor that enters the environment and that will spread more and more in the packaging, so the critical moisture content of the product will soon be reached, and the product shelf life will be shorter. The surface area of the packaging used to package the product is 0.0166 m\textsuperscript{2} [10].

The solid weight per package is obtained by correcting the overall weight with the initial moisture content of the Bolu Cukke product. Each package consists of 6 pieces, each weighing 6 grams so that the total weight is 19 grams. The solid weight is 70.77 % x 19 gram = 134.76. The pure vapor pressure in the storage room (temperature 30°C) based on the water vapor table of Labuza is 31.824 mmHg [8].

3.3. Shelf life of bolu cukke products
One of the acceleration methods that are widely applied to food products is the critical water content approach. In this method, the product is stored in environmental conditions that have relatively extreme humidity so that the product has decreased quality due to moisture absorption. This method requires the mathematical equations as a supporting instrument to quantitative descriptive of the system consisting of products, packaging materials, and environment. The Labuza equation can integrate packaging permeability elements, product’s dry weight, packaging material area, water vapor pressure difference, and isothermic sorption curve well. Based on the parameters obtained previously, the shelf life can be determined using the Labuza equation [2,7].

4. Conclusions
The occurrence of desorption or the process of releasing material moisture into the environment is the main parameter that causes a decrease in the quality of the Bolu Cukke product. A Bolu Cukke product packed in parchment paper has a shelf life of 7 days.

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