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Shelf life study of *Bolu Cukke* using the Accelerated Shelf Life Testing (ASLT) method

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Abstract. The aim of this study was to determine the shelf life of Bolu Cukke in glazed paper packaging using the Accelerated Shelf Life Testing (ASLT) Method. Determination of shelf life using the ASLT method by showing the critical moisture content and equilibrium content in Bolu Cukke. The results of critical moisture content and equilibrium content of Bolu Cukke indicate a number 2 indicating that it is at a critical point, shelf life is 6 days, with critical water content as much as 23.49% while the results using a 7-day labuza calculation showed no significant difference by storing it using critical water content. The results of the equilibrium value of 24.606% by producing a slope b value of 0.307 using the Henderson isothermic absorption curve with an MRD value of 3.1915. Estimation of shelf life of Bolu Cukke products using the labuza equation shows that the shelf life for Bolu Cukke products is 7 using glassine paper packaging which to protect the product from sunlight, air, and contamination of microorganisms. The change affected physically, chemically and microbiologically, which makes bolu cukke can no longer be consumed.

1. Introduction

Bolu Cukke is one of the traditional Bugis cakes found in the South Sulawesi region, especially in the Soppeng Regency. Bolu Cukke is known as a cake made from flour, rice flour and brown sugar which is processed by baking. Known for its good and soft taste, Bolu Cukke is favored by many people, and thus, the producers should be keen on making the cake more appealing to the consumers. In this case, the packaging is among the ways to give an attraction to the cake as well as extend its shelf life [1].

Glassine paper is one of the packaging papers that have some advantages, namely resistant to oil and grease. Thus, it is suitable for products that have excess oil content so that the oil in the product can be absorbed. As an addition to the glassine paper, the plasticizer and antioxidant are also essential complementary materials in the package, so that the packaging may perfectly fit Bolu Cukke.

This study aimed to determine the shelf life of Bolu Cukke in glassine paper package using the Accelerated Shelf Life Testing (ASLT) method, the same method used by Mariyati et al., [2] in estimating shelf life of spaghetti sauce. In the process of using parameters with environmental conditions that can accelerate the decline in the quality of a product, the ASLT is done based on the critical moisture content approach. The advantage of the method is that it has a relatively short testing



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time with good precision and accuracy [3]. So, that the shelf life obtained can be listed on the label design of the Bolu Cukke package.

2. Methodology

This research was conducted in March-May 2019 at the Laboratory of Microbiology and Food Biotechnology, Chemical Analysis, and Food Quality Supervision, Food Science and Technology, Department of Agricultural Technology, Research Center of Hasanuddin University, and Makassar Medical Laboratory Center. The equipment used in this study were cups, ovens, tongs, mixers, modified jars (humid chambers), analytical scales, thermometers, bunsen, glassine paper, duct tape, distilled water, plasticine, aluminum foil, labels.

The materials used in this study were Bolu Cukke cake, Sodium Hydroxide (NaOH), Magnesium Chloride Hexahydrate (MgCl₂.6H₂O), Potassium Carbonate (K₂CO₃), Sodium Chloride (NaCl), Potassium Chloride (KCl), Potassium Iodide (KI).

2.1. Measurement of initial moisture content (mi) moisture analyzer [4]

The moisture analyzer used for this study must be calibrated first. The moisture analyzer is firstly turned on, then lid on the device is opened, so the display status will change. Then insert the empty aluminum pan that has been cleaned, and make sure the pan is in the correct position. Close the lid again, the tool will tare automatically. Weigh the sample as much as ± 0.5 g, flatten the sample on the pan then close the device again. The moisture analyzer will heat the sample until it shows the value of the water content to a constant (3 - 5 minutes). After that, the results of the water content will come out.

2.2. Critical moisture measurement (Moisture Critical, Mc)

Samples in glassine packaging were stored in HC using a saturated NaCl solution of RH 76%. Every 24 hours, the acceptance level examination was carried out by the panels of 15 people towards the sample appearance and calculated the average acceptance test score until it reached a score of 2 (dislike). This value was then determined and stated that the product was in a critical condition. Then the measurement of water content was carried out as in point 2.1.

2.3. Water content equilibrium (Moisture Equilibrium, Me) [5]

The prepared saturated salt solution in HC (Table 1). 5 grams sample in a package was placed by hanging it in HC. Every 24 hours, it was weighed until it reached a constant weight (balance water content). Then the water content was measured using the oven method. Furthermore, the sorption isotherm curve was made by plotting the value of water content and equilibrium water activity.

Table 1. Saturated salt solutions and RH values

No.	Kind of Salt	RH (%)	Quantity	
			Salt (gram)	Water (mL)
1.	NaOH(H ₂ O)	7	150	85
2.	MgCl ₂	32	200	25
3.	K ₂ CO ₃	43	200	90
4.	KI	69	200	50
5.	NaCl	76	200	60
6	KCl	84	200	80

2.4. Determination of the isothermic sorption model [6]

The balance of water content (M_c) together with aw, was included in the isothermic sorption equation model of Chen Clayton, Henderson, Hasley, Caurie, and Oswin. The five models of sorption isotherms

were evaluated through the value of **1** Mean Relative Deviation (MRD). If the **1** MRD value is <5, the sorption isotherm model could describe the actual or very precise condition. If 5 <MRD <10 then the model was rather precise in describing the actual situation, and if the MRD > 10 then the model does not accurately describe the actual condition.

2.5. Determination of supporting parameters

The packaging permeability value ($\frac{k}{x}$), was obtained from the reference library. The value of saturated vapor pressure (Po) at 30°C was obtained from the Labuza table. The value of b (curve slope) was obtained from the gradient curve of the selected isothermic sorption equation model. The value of the cross-sectional area (A) was obtained by multiplying the dimensions of the packaging. The total solids value (Ws) was obtained by correcting the overall weight of the sample reduced by the initial moisture content.

2.6. Shelf life estimation [7]

All parameters measured and set in the previous stage, among others: **3** Mi, Mc, Me, k/x, Po, b, A and Ws were integrated into the Labuza equation (1982), as follows

$$\theta = \frac{\ln \frac{(M_e - M_o)}{(M_e - M_c)}}{\frac{k}{x} \left(\frac{A}{W_s} \right) \frac{P_o}{b}} \tag{1}$$

Notes:

- Θ = Estimated shelf **5** life (days)
- Me = Balanced wa**5** ter content of the product (g H₂O/g solid)
- Mi = Initial water content of the product (g H₂O/g solid)
- b = Slop isothermic sorption curve
- Mc = **5** atitial water content (g H₂O/g solid)
- $\frac{k}{x}$ = Permeability of bottled water (g/m².day.mmHg)
- A = packaging surface area (m²)
- Ws = dry weight of packaged product (g solid)
- Po = saturated vapor pressure (mmHg)

3. Result and discussion

3.1. Bolu Cukke

Bolu Cukke is a bakery product made from flour, sugar, fat and eggs. Making Bolu Cukke requires the development of gluten, chemical development materials, and the formation of complex emulsions in oil where the air layer consists of dissolved sugar and dissolved flour particles. Bolu Cukke product is a typical Bugis food originating from Soppeng regency. The dominant taste of Bolu Cukke is brown sugar because brown sugar is the main ingredient as well as the hallmark of the product. The main difference between Bolu Cukke and bread products is the dough texture, thick textured Bolu dough [8].

3.2. Estimation of shelf lifex

Changes to food products are **15** factor that shortens the shelf life, and among the basic things in determining the critical point of shelf life. The shelf **23** itself can be determined by finding out the critical point of the product because the critical point is one of the most sensitive factors to discover the shelf life of the product. In this case, the semi-trained panelists may find out the critical point by

conducting organoleptic tests. The score of two (2) indicates that the product is no longer acceptable to the panelists so that it can be said that the product has been at a critical point. According to [9], the expiry criteria of some food products can be determined using a critical point reference.

3.3. Moisture Content and Critical Moisture Content

Moisture content is one of the contents contained in the product ingredients, which shows how much water is in the material, whereas the critical moisture content is the water content in the product which is interpreted as a condition where the product has arrived at the limits of organoleptic consumer acceptance. The consumer's measurement procedure to determine the critical moisture content is organoleptic testing. The organoleptic testing of Bolu Cukke requires the tests on aroma, texture, and color by providing a hedonic assessment, which consists of a score of 1 to 5 where a score of 1 (very not likes), score 2 (dislike), score 3 (rather like) score 4 (like) and score 5 (really like). If the panelists give a value of 2 on average then we can safely say that the product has been placed at critical water content. This is consistent with the statement of [10] who says that the critical moisture content is the water content when the organoleptic sample is no longer acceptable to consumers. The acceptance of the length of storage is shown in figure 1.

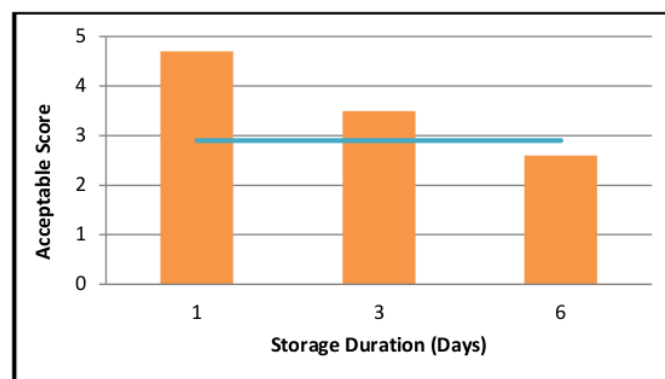


Figure 1. Acceptance of the length of storage

Figure 1 shows that during the first day to the sixth, the organoleptic scores have been decreasing in which the average first-day organoleptic results were 4.8, the third day was 3.5 and the sixth day was 2.6, indicating that Bolu Cukke was already in a critical condition as the results are now in the value of 2. After knowing the product of Bolu Cukke has been at a critical point, then the calculation of critical moisture content is performed. The final calculation shows that the critical moisture content of Bolu Cukke is 0.2349% BK. If we associate this result with the initial moisture content of 0.2215% BK, it can be concluded that there is an increase in the amount of water content in Bolu Cukke products indicated that there had been a process of water absorption from the environment into the product that caused an increase in the weight of the moisture content. This corresponds with the statement of [10], which states that the increase in RH will be followed by an increase in moisture content and affect product quality.

3.4. Equilibrium moisture content

Equilibrium moisture content is the water content in a balanced condition which does not decrease and increase again in the product for a long period of time. This is in accordance with [11] who states that the equilibrium moisture content is the water content of material after being in on its environmental condition for a long period of time. To determine the equilibrium moisture content, this study stores Bolu Cukke in six Humidic chambers containing various types of saturated salt solutions with

different RH. It aims to represent the overall range of water activity and to obtain the smoothest and the most precise isothermic absorption curve for determining the product’s shelf life.

The results showed that the time to achieve the equilibrium state in Bolu Cukke is starting from the lowest RH to the highest RH sequentially, 7% RH with equilibrium moisture content value of 3.57%, RH 32% moisture content 9.97% RH 43% moisture content 13.97%, RH 69% moisture content 19.80%, RH 76% moisture content 23.57%, and RH 84% moisture content 28.06%. This shows that the lower the RH, the faster the time for the product to achieve the equilibrium, and the lower the product’s equilibrium moisture content. These results indicate that the higher the RH in storage, the longer the process of diffusion of water vapor toward equilibrium and vice versa, the lower the RH in storage, the faster it goes to equilibrium. This is consistent with the statement of [12] which states that the closer the aw value of the product to the RH environment, the shorter the time needed to achieve equilibrium. The process that occurs during the storage with different RH is the process of absorption and desorption, where absorption is the process of water vapor absorption by the material from the environment and desorption is the process of releasing water vapor into the environment.

Table 2. Equilibrium Moisture Content in Various Solution

Solution	Aw (Standar)	Me Value
NaOH (7%)	0.07	3.57%
MgCl ₂ .6H ₂ O (32%)	0.032	9.97%
K ₂ CO ₃ (43%)	0.43	13.97
KI (69%)	0.69	19.80 %
NaCl (76%)	0.76	23.57%
KCl (84%)	0.84	28.06%

3.5. Isothermal Sorption Curve

The isothermic sorption curve is the relationship between equilibrium moisture content which plotted with the value of water activity in the storage room and the moisture content per gram of material. The equilibrium moisture content value of each storage RH in the solution can be made an isothermic sorption curve. The isothermic sorption curve has five mathematical equation models, namely Chen Clayton, Henderson, Hasley, Caurie, and Oswin. This is in accordance with [13] who states that an isothermal adsorption curve is a curve that describes the relationship between water activity (aw) or equilibrium relative humidity (RH) in a storage room (ERH) with water content per gram of food.

Table 3. MRD Calculation of Isothermic Sorption Models

Model	Formula	MRD Value
Chen Clayton	$\ln(1/a_w)=1.30-11.0Me$	6.257
Henderson	$\log [\ln(1/(1-a_w))]=1.134+17.8 \log Me$	3.1915
Hasley	$\log [\ln(1/a_w)]=-1.34-1.29 \log Me$	15.57
Caurie	$\ln Me=-3.28+2.492 a_w$	13.71
Oswin	$\ln Me=-2.35+1.374 \ln [a_w/(1-a_w)]$	33.24

The calculations of the five isothermic sorption models each results in MRD (Mean Relative Deviation) values, which are a measure of the accuracy between the equilibrium moisture content resulted from the experiments with the isothermic sorption model. Table 03 shows that the Henderson model has the smallest MRD value, with a value of 3.1915. Although the model on the other curves has a high average value, the Henderson model is the model that produces the right isothermic absorption curve because the MRD value is <5. In determining the value of isothermal

sorption curves, there are two models that almost have the same value, namely Chen Clayton as much as 6.257% and Caurie value as much as 3.1915%, Since Henderson value is the lowest, then it is taken in determining the isothermic sorption curve because the smaller MRD value, the more appropriate the model. This is consistent with [14] which states that the smaller the MRD value, the more appropriate the model is in describing the phenomenon of isothermic absorption that occurs.

The figure shows the isothermic absorption curve resulted from the experiment coincides with the Henderson isothermic absorption curve and has the lowest MRD value of 0.915%. This means that the phenomenon of isothermic sorption is evident. The results of the relationship between A_w and the equilibrium moisture content produce a linear equation $y = a + bx$ or $y = 0.0066 + 0.307x$ so that the slope value (b) of the isothermic sorption curve on Bolu Cukke is 0.307.

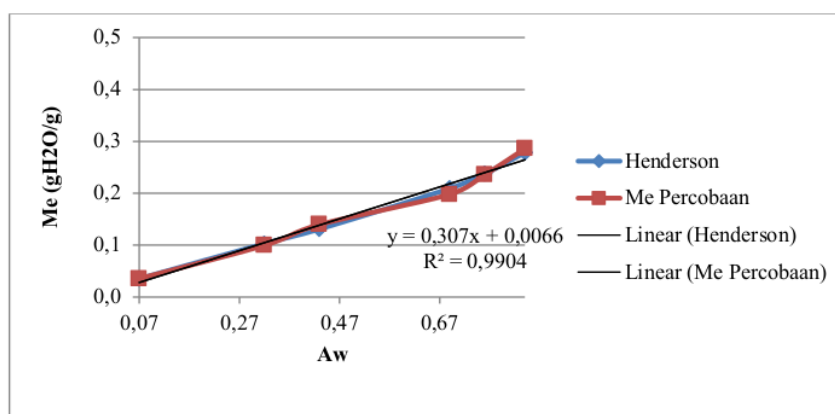


Figure 2. The isothermic absorption curve resulted from the experiment coincides with Henderson's.

3.6. Supporting parameters

There are several things that become supporting parameters in determining the shelf life of the product, namely packaging of the product, permeability (k/x), pure vapor pressure on storage (P_0) and surface area of packaging (A). These are all supporting parameters in estimating the shelf life of Bolu Cukke product. Its permeability, surface area, and pure vapor pressure can be determined by calculating the Labuza equation.

Paper packaging that is covered by plasticizer additives makes this package difficult to be attacked by microbes in the environment due to the important role of the packaging's permeability. Permeability on the packaging protects the package from air and water vapor in which if the permeability is high, then more air and water vapor enters, and if the permeability is low, air and water vapor are less likely to enter the package. The permeability of the packaging of this plastic glassine paper is 1.1 gH₂O/m². This is in accordance with [12] who states that the higher the permeability value of the package (k/x), the greater the diffusion of water and gas through the package so that the product's moisture content is greater.

The surface area of the packaging is also one that affects a product's shelf life, where the determination of the size of the packaging area is done by multiplying the length and width of the packaging used. The wider and longer the packaging in the product, the greater the opportunity the water vapor spread on the package, which makes the critical moisture content in the product will be achieved. According to [15] the wider the surface area of the packaging, the higher the water vapor entering the environment. The water vapor will be spread more widely in the package so that the

critical moisture content of the product will soon be reached. The surface area of the packaging used to package the product is 0.0170 m².

This shelf-life calculation is a mere estimation because the product’s damage was analyzed solely based on water absorption until the critical conditions are reached. Thus, it is necessary to calculate shelf life based on other critical qualities such as free fatty acids, microbial calculations, and peroxide numbers so that the shelf life obtained could be more accurate.

3.7. Calculating the estimated shelf life

Shelf life is among the most important things that consumers need to know about food products they want to consume. They can do this by looking at the information on the time period on the product label. In addition to that, the consumers can also see important components of the product such as paying attention to changes in safety, nutrition, physical and organoleptic properties. According to [5], shelf life information is a form of guarantee for food producers to consumers that the product has a good quality and is safe for consumption before the specified expiration date has been reached.

Table 4. Parameter Values for Calculation of Shelf Life of *Bolu Cukke*

Parameter	Value
Initial Moisture (Mi) gH ₂ O/g solid	0.2215
Critical Moisture (Mc) gH ₂ O/g solid	0.2349
Ishotermic Slope (b)	0.307
Equilibrium Moisture (Me) gH ₂ O/g solid	0.2460
Packaging Permeablity gH ₂ O/m ² .day.mmHg (k/x)	1.1
Packaging Size (m ²)	0.0170
Dry Weight (gram)	18.225
Saturated Vapor Pressure in 30 ^o C (mmHg)	31.824

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

$$\theta = \frac{LN \frac{(0.24606-0.2215)}{(0.24606-0.2349)}}{1,1 \left(\frac{0.0170}{18.2215} \right) \frac{31.824}{0.307}}$$

$$\theta = \frac{LN 2.2072}{0.1063}$$

$$\theta = \frac{0.7917}{0.1063}$$

$$\theta = 7.4422 = 7 \text{ days}$$

The results above indicate that there is a change in circumstance, food value, quality and distribution of the product at the time of storage. It can be concluded that the longer the storage of the product, the more changes in the product, both the nutritional value or physic of the product. This is in accordance with the statement of [16] which states that when newly produced, the quality of the product is considered to be 100%, and will decrease along with the duration of storage or distribution. During storage and distribution, food products will lose weight, food value, quality, value for money, growth power, and trust.

The values integrated into the Labuza equation show that the shelf life of the Bolu Cukke product is 7 days. These results indicate that the Labuza calculation does not significantly differ with the calculation of critical moisture content in which the results only differ a day on the store of critical moisture content. This can be influenced by-products that are wet basis > 18% which results in no significant difference from the results of critical moisture content. It can be concluded that to find out the estimated shelf life, we must first recognize the key parameters of the product.

4. Conclusion

The estimation result of the shelf life of Bolu Cukke product using the ASLT method with the critical moisture content approach of the labuza calculation lasted for 7 days.

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