11. Shelf-life Prediction of Soneca Using Accelerated Shelf-life Tests Approach to Critical Water Levels

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Shelf-life Prediction of Soneca Using Accelerated Shelf-life Tests Approach to Critical Water Levels

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ABSTRACT

Shelf life is information that must be included in food product packaging. Estimated shelf life of food products can be done conventionally (Extended Storage Studies) and acceleration (Accelerate Shelflife Testing). Estimation of shelf life by ASLT can be done using the critical moisture content approach, especially for products easily damaged by water absorption. The purpose of this study was to determine the shelf life of Soneca biscuits packaged using three different packages, namely Polypropylene (pp) plastic packaging, Metalized Plastic, and Aluminum foil. The method of determining the shelf life used in this study uses the ASLT method with a critical moisture content approach. Based on the research, it is known that the critical moisture content of biscuits is obtained when the product moisture content reaches 0.0812 (g H2O/g). The isothermic sorption curve of the biscuits obtained can be appropriately expressed using the Hasley model equation. Estimation of shelf life was carried out under storage conditions at 30 ºC and 75% RH. The study results show that the predicted shelf life of biscuits with metalized plastic VM-PET packaging is 17.76 months, with aluminum foil packaging is 19.85 months and using PP plastic packaging is 1.72 months. Therefore, it is known that the best shelf life is the use of aluminum foil packaging.

Keywords: Biscuits, pedada, mocaf, and isothermic sorption curve

Introduction

Biscuits are one of the most popular foods in Indonesia. Many studies have been investigated on laboratory-scale biscuits, both in the context of reformulation and formulation of new products (Calligaris et al., 2016). One of the studies that have been carried out is the formulation for people with diabetes, which was later known by the trademark, Soneca. Soneca Biscuits are characterized by their moisture content and crunchy texture. Products with low moisture content, such as biscuits, are prone to loss of crispness and are sensitive to moisture absorption. Prolonged direct exposure to dry products under ambient storage conditions causes water absorption from the atmosphere into the product matrix, causing it to lose its crispness and become soft, mushy, and consequently less acceptable to most consumers (Putri et al., 2021). Expiration criteria on dry food are on the absorption mechanism of water vapor (Tristantini et al., 2019). The leading cause of physical damage to biscuit products is the loss of crispness in biscuits.

To be able to maintain biscuit products with low water content, packaging can be used as an effort to preserve the product. There are many types of biscuit packaging found in the market, including plastic packaging, aluminum foil, and metalized plastic (Forsido et al., 2021). In plastic packaging, generally, two types of plastic are often used as food packaging, namely Polypropylene (PP) plastic and Polyethylene (PE) plastic (Namira et al., 2020). In addition to low water vapor

permeability, PP plastic packaging is the safest type of packaging for food compared to other types of plastic packaging (Tristantini et al., 2019). Meanwhile, aluminum foil is a hermetic packaging, opaque, flexible, and can be used as a coating or reinforcement material coated with plastic or paper (Marsvia et al., 2020). Metalized plastic is a good and inexpensive alternative to food packaging. A thin layer of aluminum foil enhances the barrier properties against moisture, air, and odors.

Different types of packaging will have different responses to the product. For example, research on estimating the shelf life of food products with different types of packaging using the critical moisture content approach has been carried out several times, including that the shelf life of soft dough biscuits and complex dough products packaged with metalized plastic is 17.4 months and 16.5 months, respectively (Marsvia et al., 2020). On the other hand, the shelf life of biscuits based on protein concentrate and spirulina with PP plastic packaging and Retort pouch were obtained, respectively, namely 23.6 months and 2.8 months (Ganje et al., 2016). Then the estimated shelf life of pineapple cookies with metalized plastic packaging is 400 days, longer than that of Polypropylene (PP) plastic packaging, which is 97 days (Afifah & Ratnawati, 2021).

Estimated shelf life of food products can be done conventionally (Extended Storage studies) and accelerated (Accelerated Shelf-life Testing). The conventional shelf life estimation is done by storing the product in daily conditions until it reaches the expiration quality level. This method requires a long time and expensive analysis costs but is very accurate and precise. At the same time, the acceleration method can be done in a shorter time with reasonable accuracy (Latief et al., 2020). The acceleration method can be carried out using the Arrhenius model approach and the critical moisture content model. The Arrhenius model is used for products sensitive to changes in storage temperature. In contrast, the critical moisture content model is used for easily damaged products due to water absorption from the environment during storage (Syahrul et al., 2020). Therefore, the application of the acceleration method needs to pay attention to the characteristics and causes of product damage that will determine its shelf life.

The information regarding the correct type of packaging and the estimated shelf life of Soneca biscuits is not yet known. Given the importance of the information value of a food product's shelf life, it is necessary to research estimating the shelf life of pedada fruit biscuits and mocaf flour using three different types of packaging using the ASLT (Accelerated Shelf-life Testing) method with a critical moisture content approach.

Material and Methods

Sample of termite nest was collected in September 2016 from Pananjung Pangandaran Nature Reserve, West Java, Indonesia. Termite nest was obtained by cruise method. The type of collected nest termite was carton nest. Termite nest samples (200 g per sample) were placed in polyethylene bags and immediately transported to Microbiology Laboratorium of Research Center for Biomaterials- LIPI. Termites sample (worker and soldier) inhabiting the nest were collected and preserved in 70% alcohol tube. The termites were identified based on key identification. Photographs were taken with digital microscope with 40 – 80 × magnification. Nest samples were ground into fine particles and air-dried at an ambient temperature for 7 days before the isolation of Actinomycetes.

The ingredients used in making pedada flour biscuits and mocaf flour include pedada fruit while cassava. Other supporting ingredients include skim milk powder, salt, refined sugar, margarine, egg yolks, glucose syrup, sodium stearoyl lactylate (SSL), and sodium bicarbonate. Chemicals used in the research to estimate the shelf life of biscuits include salt to make a saturated salt solution including Aquades, (CH_3COOK, $MgCl_2$, K_2CO_3, $NaNO_2$, $NaCl$, KCl, $BaCl$, and KNO_3), silica gel (Merck), and vaseline.

The tools used in manufacturing biscuits include a basin, analytical scale, pot, spoon, 80 mesh sieve, stove, cabinet, blender, biscuit mold, oven. Tools used in estimating shelf life include glass jars (modified desiccators), wire ram supports, closed plastic containers, silicone glue, cups,

weighing bottles, drying ovens, metal clamps, desiccators, mortar, hand sealers, texture analyzers, electric stoves, and glassware. There are eight modified desiccators used, each containing a different saturated salt solution. The desiccator containing the saturated salt solution was left for 24 hours without being filled with the sample to create an equilibrium RH condition. There are three different types of packaging, namely PP plastic, aluminum foil, and metalized plastic.

This research was carried out through two stages; the first stage was the manufacture of Soneca biscuits with the formulation obtained from the previous research treatment by Maria (2018), namely 10 grams of pedada flour, 90 grams of mocaf flour, 20 grams of skim milk, 30 grams of refined sugar, 1 gram of salt, margarine 50 grams, egg yolks 8 grams, SSL 0.5 grams, sodium bicarbonate 0.5 grams, glucose syrup 20 ml. Then the estimation of shelf life by measuring the parameters of estimating the shelf life of biscuits using the ASLT method with a critical moisture content approach based on the Labuza equation (Putri et al., 2021). Estimation of shelf life is based on the critical moisture content approach. The parameters that must be determined in estimating shelf life using the ASLT method of critical water content approach include:

Initial water content measurement (Mi)

The initial moisture content (Mi) of pedada and mocaf biscuit products was determined by measuring the moisture content (g H2O/g solids) using the oven method (Horwitz & Latimer, 2005).

$$\text{water content bk} = \frac{\text{Starting Material Weight} - \text{Dry Material Weight}}{\text{Dry Material Weight}} \times 100\%$$

Determination of critical water content (Mc)

The critical moisture content (mc) was determined by storing the sample for 0, 30, 45, 60, 90, and 120 minutes without packaging in a closed container, equipped with a buffer and filled with 2 L of water so that the decrease in crispness was faster where the RH of water was 100% equivalent to aw = 1. This process produces a series of samples with different storage times. Finally, samples were repackaged for immediate organoleptic testing of 30 untrained panelists with texture/crispy parameters and water content measurements (Tristantini et al., 2019).

Determination of equilibrium moisture content (ISA curve)

The first thing to do is prepare a saturated salt solution. According to (Anggreni *et al.*, 2021), the salt used can cover the range of water activity values between 0.20 and 0.95. The salts used are CH_3COOK, $MgCl_2$, K_2CO_3, $NaNO_2$, NaCl, KCl, BaCl, KNO_3. The activity of water and RH resulting from the saturated salt solution can be seen in table 1. First, the saturated salt solution is put into a desiccator. Then closed and left for 24 hours at room temperature conditions. A total of 2 g of samples have been mashed, and the water content is known to be placed in a cup of known weight. Next, the cup containing the sample is put into a desiccator which already contains a saturated salt solution. The desiccator was then stored at average room temperature (30 ±2 °C). The samples and plates were then weighed periodically every 24 hours until a constant weight was obtained, which means that the equilibrium moisture content (Me) has been reached. The constant weight was indicated by the difference between three consecutive weighing days of not more than 2 mg/g for samples stored at RH below 90% and not more than 10 mg/g for samples stored at RH above 90% (Tristantini *et al.*, 2019). After obtaining a constant weight of the sample, the moisture content was measured using the oven method (AOAC, 2005). Next, a water sorption isotherm curve was made based on the sample's equilibrium moisture content (Me) at various RH values.

Model accuracy test

The MRD value is used to test the accuracy of the model by using the formula.

$$MRD = (100 \sum_i)/n=1 \ (Mi-Mpi)/Mi$$

MRD value < 5, then the isothermic sorption model can describe the actual situation very accurately. If 5 < MRD < 10, then the model is entirely appropriate to describe the actual situation, and if MRD > 10, the model is not appropriate to describe the actual condition. The selected model is used to determine the slope value of the isothermic sorption curve.

Determination of supporting variables

The area of packaging (A) used is calculated by multiplying the length by the width of the package in m^2.

A = Length x width

The weight of solids per package (Ws) is expressed as g solids per package and is calculated based on the formula:

% solids   = (1-wet basis moisture content) x 100%
Weight of packaging solids (g)   = Weight of sample per pack (g) x % solids

The packaging used in this research consists of three types of packaging, namely Polypropylene (PP) plastic, Metalized plastic, and aluminum foil. The permeability value of PP plastic packaging is 0.0785 g/m2.hari.mmHg, the permeability of Metalized plastic VM-PET packaging is 0.0076 g/m2.hari.mmHg) and the permeability of Aluminum foil packaging is 0.0068. Saturated vapor pressure is determined based on the table of saturated vapor pressure at a temperature of 30 ºC (Anandito et al., 2017).

Shelf-life calculation

$$t = \frac{\ln(\frac{me - mi}{me - mc})}{\frac{k}{x}(\frac{A}{Ws})\frac{Po}{b}}$$

Where:

t   = estimated shelf life (days)
Me   = equilibrium water content of the product
Mi   = initial water content of the product
b   = slope of isothermic sorption curve
Mc   = critical water content
k/x   = vapor permeability of packaged water
A   = surface area of the packaging
Ws   = dry weight of the packaged product
Po   = saturated vapor pressure

Results and Discussion

The initial moisture content of Soneca biscuits determined using the oven method is known to be 0.0347 g H_2O/g solid (Horwitz *et al.*, 2005). Critical water content is when food products begin to experience a decline in quality so that they are no longer accepted by consumers organoleptically. The critical moisture content (Mc) was determined by organoleptic analysis (hedonic test) on seven biscuits stored for different storage periods. Then in each series of samples, water content was measured using the AOAC oven method (2005). The critical moisture content or MC of the product can be determined through a linear equation obtained from the regression results

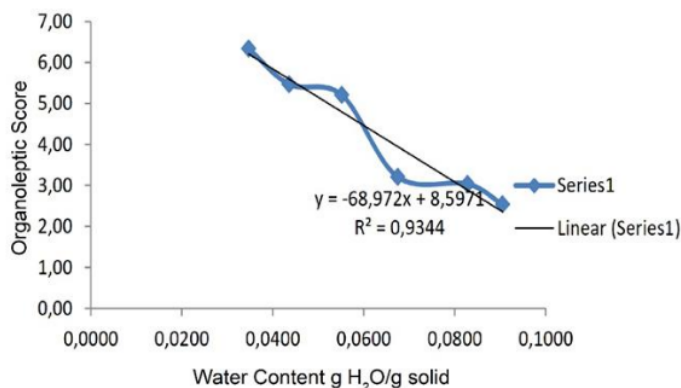of the logarithmic relationship curve between the water content data and the organoleptic texture score.

Figure 1. Correlation curve of texture organoleptic value with water content

The linear regression equation obtained is y = -68.9720 x + 8.5971 (can be seen in Figure 1). The critical water content of the product is obtained by substituting the number 3 in the obtained linear regression equation (Y=3). This shows that the product's condition is considered to have been rejected by consumers, and this condition must be watched out for to ensure customer satisfaction and comfort (Syahrul et al., 2020). From this calculation, the critical moisture content (Mc) of Soneca biscuits is 0.0812 H_2O/g solid, which means that when the moisture content is reached, the biscuits are in a critical state. The critical water content that is different for each product can be caused by the processing process, product shape, size, and composition specific to each product. According to (Wahyuni et al., 2018), wheat flour has a higher water absorption capacity than other flours. The critical water content value of products made from wheat flour can obtain a lower critical water content than other dry products.

Equilibrium moisture content and isothermic sorption curve

The equilibrium moisture content required to construct an isothermic sorption curve is obtained by conditioning the product in several saturated salt solutions with relative humidity (Putri et al., 2021).

Table 1. Equilibrium moisture content (Me) of pedada fruit biscuits and mocaf flour

Mineral Salt	ERH (%)	Aw	Me
CH_3COOK	22.5	0.225	0.0522
$MgCl_2$	32.4	0.324	0.0667
K_2CO_3	43.2	0.432	0.0802
$NaNO_2$	64.3	0.643	0.1082
NaCl	75.1	0.751	0.1574
KCl	83.6	0.836	0.2254
BaCl	90.3	0.903	0.3194
KNO_3	92.3	0.923	0.3802

During storage under different RH conditions, there will be interactions between the biscuit product and its environment. During storage of the product in various saturated salt solutions, a water equilibrium process occurs. Namely, the product absorbs water from the environment (adsorption) and water from the material (desorption). The adsorption process occurs at high RH, and conversely, the desorption process occurs at low RH. The transfer of water vapor is caused by differences in relative humidity so that water vapor moves from high RH to low RH (Putri et al., 2021). This process will cause an equilibrium between the aw of the product and the aw of the chamber environment. Equilibrium moisture content (Me) is the water content when the product water vapor pressure is in equilibrium with the environment (constant product weight) (Tristantini et al., 2019). The equilibrium moisture content will describe the properties of the product material to water. Water sorption isotherms explain the relationship between the water content of the material and the equilibrium relative humidity (RH) at a specific temperature. This curve illustrates the hydration properties of foodstuffs, namely the ability of foodstuffs to naturally absorb water from the surrounding air and can otherwise release some of the water they contain into the air (Latief et al., 2020).
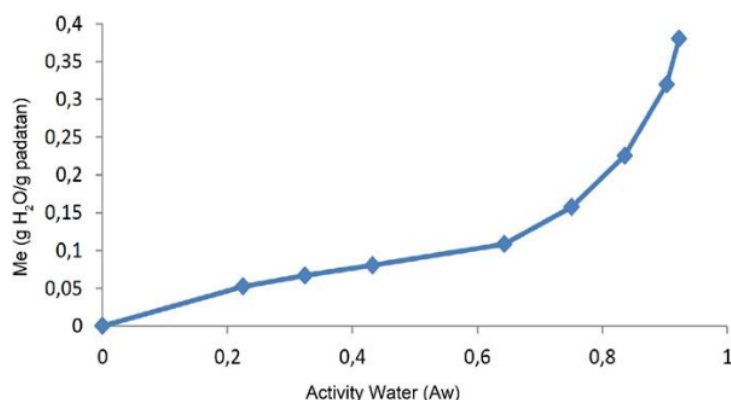

Figure 2. Biscuit isothermic sorption curve

Isothermal sorption of foodstuffs is needed to determine the quality, stability, and shelf life of foodstuffs. Soneca biscuit adsorption isotherm curve produces a curve that follows the sigmoid shape, namely the shape of the water absorption isotherm curve in the type II form. According to (Merkx et al., 2021), Type 2: sigmoidal sorption isotherm, where the curve is concave upwards, takes into account the presence of multilayers present on the internal surface of a material. This sigmoid shape is caused by the capillary effect and the interaction between the surface of the material and water molecules. In general, the shape of the product isothermic sorption curve will have a distinctive shape for each type of food material. The equilibrium moisture content used in determining the shelf life of food products is at a certain RH and in the equation of the selected isothermic sorption curve model. In this study, the equation of the isothermic sorption curve used is Hasley, Chen – Clayton Henderson, Caurie Oswin, Gab. The accuracy and smoothness of the isothermic sorption curve model in describing the sorption phenomenon are determined based on the increasing closeness of the isothermic sorption curve from the equation model to the experimental isothermic sorption curve. This study made a comparison between the experimental water content and the model water content through MRD calculations. The MRD value of each model can be seen in table 2.

Table 2. MRD values for each model of the isothermic sorption equation

Model	MRD
Hasley	3.0900
Chen-clayton	27.2625
Henderson	14.1375
Caurie	13.6688
Oswin	6.3863
GAB	3.5413

The equation of the selected model can be seen from the smallest MRD value (Latief et al., 2020). If the MRD value <5, then the sorption isotherm model can describe the actual situation or is very precise. If the MRD value is between 5 to 10, then the model is entirely appropriate, and if the MRD value is > 10, the model is not appropriate to describe the actual situation. In this study, the Hasley equation has the MRD with the smallest value. The Hasley model equation provides an expression for the condensation of multilayers at a relatively large distance from the surface, assuming that the potential energy varies as an inverse power of n from its distance from the surface (Tristantini *et al.*, 2019). This equation is a good representation of the adsorption data regarding type I, II, or III isotherms. In addition, this equation explains the absorption behavior of food products containing starch. The isothermic sorption curve equation of the Hasley equation for Soneca flour biscuits is Log (ln (1/aw) = Log -1.7274-1.4867 log Me. The selected equation can also be used to determine the equilibrium moisture content used in calculating the shelf life estimation. The selected equation can be used to determine the slope of k.
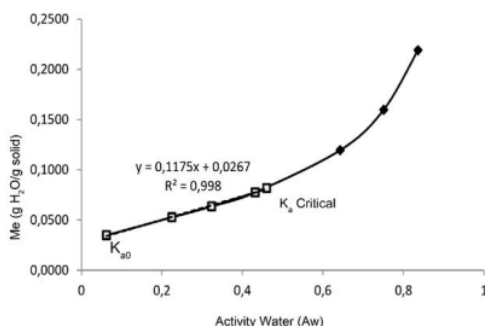

Figure 3. The slope of Biscuit Isothermic Sortion Curve Using the Hasley Model Equation

The linear area to determine the slope of the isothermic sorption curve is taken between the initial moisture content and the critical water content on the selected model equation curve (Latief *et al.*, 2020). In this study, the determination of the slope (b) value was obtained by plotting the value of the equilibrium moisture content with the relative humidity (RH) of the Hasley model. The results of the linear regression of the sorption isotherm curve resulted in the line equation y = 0.1175x + 0.0267 (R^2 = 0.998). Based on this equation, the value of the b (slope) curve is 0.1175.

Supporting variables for estimating shelf life

This is based on the average temperature in Indonesia. The saturated vapor pressure at a temperature of 30 °C is based on the average temperature in Indonesia. The saturated vapor pressure at a temperature of 30 °C is 31.82 mmHg taken from the temperature-pressure table. The

permeability value of PP plastic packaging, according to (Deki, 2010) is known to be 0.0785 g/m^2.hari.mmHg, the permeability of Metalized plastic VM-PET packaging is 0.0076 g/m^2.hari.mmHg and the permeability of Aluminum foil packaging of 0.0068 g/m^2.day.mmHg (Anggreni et al., 2021). The packaging permeability value is obtained from secondary data. Based on the parameters for determining the shelf life above, the shelf life can be estimated using the pumpkin equation (1982). Estimation of the shelf life of Soneca biscuits in three different types of packaging can be seen in table 3.

Table 3. Results of calculation of shelf life on various packages

Parameter	Unit	PP Plastic	*Metalized plastic*	Aluminum m foil
Product Initial Moisture Content	g H_2O/g solid	0.0347	0.0347	0.0347
Product Critical Moisture Content	g H_2O/g solid	0.0812	0.0812	0.0812
Hasley's equation model: log(ln(1/aw) = log -1.7274-1.4867 log Me				
The slope of the ISA curve (slope)	0.1175	0.1175	0.1175	
Equilibrium water content	g H_2O/g solid	0.1592	0.1592	0.1592
Packaging permeability	g/m_2hari.mm Hg	0.0785	0.0076	0.0068
Packing area	m_2	0.0206	0.0206	0.0206
Weight of packaging solids	g Solid	48.32	48.32	48.32
Saturated vapor pressure 30 ᵒC	mmHg	31.82	31.82	31.824
Shelf-life	Day	51.59	532.53	595.53
Shelf-life	Month	1.72	17.76	19.85

Typically packaged biscuits usually have a shelf life of 6 months, but the actual shelf life of the product tends to be much larger (Calligaris et al., 2016). The type of packaging will significantly affect the shelf-life value because each packaging material has a different ability to protect the packaged product. Based on table 13, it can be seen that the use of packaging significantly affects the shelf life. Estimation of shelf life of pedada fruit flour biscuits and mocaf flour stored at 30 ᵒC and 75% RH in Polypropylene, metalized plastic, and aluminum foil packaging was known to be 1.72 months, 17.76 months, and 19.85 months, respectively. From these results, it can be seen that the shelf life of packaged products is inversely proportional to the characteristics of the permeability value of the packaging. Estimating the shelf life of biscuits using aluminum foil packaging obtained the results of the most extended shelf-life estimation compared to other packaging. PP packaging has a higher water vapor permeability when compared to metalized plastic and aluminum foil packaging. This causes biscuits with PP packaging to have the shortest shelf life. The large packaging permeability will cause the product shelf life to be shorter because the high permeability of the packaging can absorb more water vapor from the environment into the product so that the packaged product will be damaged more quickly (Kulchan et al., 2010).

Conclusion

Biscuits are one of the hygroscopic food products. It is known that the critical quality of biscuits is the loss of crispness. Consumers do not accept crunchiness at a critical water content of 0.0817 g H_2O/g solids. The biscuit isothermic sorption curve obtained is included in the type II ISA curve and can be accurately described using the Hasley model. After combining all the parameters of the estimated shelf life with storage conditions at 75% RH and a temperature of 30 ᵒC, the predicted shelf life of Soneca biscuits with VM-PET metalized plastic packaging is 18.00 months with aluminum foil packaging is 20.12 months and using PP plastic packaging is for 1.74 months. The

value of the permeability of the packaging is inversely proportional to the calculation of the shelf life obtained. So based on this research, it is known that the best shelf life is aluminum foil packaging.

Acknowledgment

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