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Formulation and Shelf Life Prediction of Cookies From Modified Cassava Flour (Mocaf) In Flexible Packaging

M Kurniadi^{1*}, Y Khasanah¹, A Kusumaningrum¹, M Angwar¹, D Rachmawanti², N H R Parnanto² and L D Pratiwi²

¹Research Unit for Natural Products Technology, Indonesian Institute of Sciences (LIPI), Yogyakarta, Indonesia

²Department of Science and Food Technology, Universitas Sebelas Maret, Surakarta, Indonesia

*Email: hm_kur@yahoo.com

Abstract. The product development from modified cassava flour (mocaf) cookies has not been done much. This research was done to find out the best formulation on the cookies from mocaf and to predict its shelf life on flexible packaging. Formula cookies using mocaf flour, egg - tempe flour, green bean flour and catfish flour, each with a ratio: F1 (63%: 5%: 12%: 15%); F2 (63%: 5%: 12%: 20%); F3 (58%: 5%: 12%: 25%). Shelf life prediction using Accelerated Self Life Time (ASLT) method based on critical moisture content. The packaging that used is polyethylene, aluminum foil and metalizing. The results of this research showed the best formula was F2 containing protein 12.06 %, and energy 511.56 kcal / 100 g. The critical water content of cookies 6.89 %. Permeability of polyethylene, metalizing and aluminum foil were 0.05 gH₂O / day.m².mmHg, 0.006 gH₂O / day.m².mmHg and 0.006 gH₂O / day.m².mmHg respectively. That packaging was able to maintain the cookies quality for 1.5 months (polyethylene), 12 months (metalizing), and 19 months (aluminum foil).

Keyword: Shelf life, cookies, mocaf, flexible packaging

1. Introduction

Today, stunting is a problem must be solved immediately in Indonesia. In 2013, stunting was approximately 37 % (9 million) Indonesian children in various income groups, predominantly having fifth rank in the world [1]. Stunting was caused by less household access (prospective parents, pregnant mothers or babies under 5 years old) to nutritious food. Based on the case, we need to find out alternatives food with high calorie, protein, and micro-nutritional substances at a low price. Cookies are an alternative product, ready to eat and accept by people of all ages [2]. The main problem to make cookies is wheat flour, where this material is imported from other countries, with gluten that can be consumed by autism. Therefore, we need usable alternatives to wheat flour, such as modified cassava flour (mocaf). Mocaf has similar characteristics and quality to wheat flour but contains low protein, so we need to add other food materials as protein sources. Some of the food materials, which may be used to increase protein content, are egg-tempe flour, green bean flour, and catfish flour [3-5].

Cookies quality is speedily reduced because it easily absorbs water content [6]. Shelf life is a very important indicator to specify quality of a food product in a specific time. Some factors that affecting shelf life are a type of product, type of packaging, a technique of packaging, temperature, and moisture



[7]. The shelf life of cookies may be specified using Accelerated Shelf Life Time (ASLT), applying environmental condition parameters accelerating the process of reduction in a quality product, giving higher shelf life temperature [8]. Shelf life prediction using ASLT method is specified by critical water content method using Labuza equation and semi-empirical approach using Arrhenius equation [6][9]. This research was conducted to find out the best formula of cookies using basic materials with mixed mocaf flour, egg-tempe flour, green bean flour, and catfish flour as well as hypothesizing shelf life using ASLT method with critical water point approach in some flexible packages.

2. Materials and Methods

2.1. Materials and tools

Materials used in product formulation are mocaf, green bean flour, catfish flour, egg-tempe flour, sugar, butter, skim milk, baking powder, mocha pasta, and water. Materials for analysis are LiCl, KCH_3CO_2 , MgCl_2 , K_2CO_3 , $\text{Mg}(\text{NO}_3)_2$, KI, NaCl, KCl, and K_2SO_4 . Packaging was used are aluminum foil, metalized, and polyethylene (PE). The tools are oven, mixer, analytical scale, cup, desiccators, clamper, mortar, and laboratory glass tools, glass jars, aluminum cup, thermometer.

2.2. Methods

This research consists of some stages : (1) making of cookies, (2) analysis: organoleptic test, chemical analysis, and shelf life predictions. The formula of cookies was in Table 1. The organoleptic test includes parameters of color, texture, aroma, taste, and overall preference with the scoring method in scale from 1 to 5, using 25 semi-trained panelists [10]. Chemical analysis of selected cookies formula (results of preference test) consists of water content (Thermogravimetry)[11], ash [11], protein (Micro Kjeldhal)[11], fat (Soxhlet) [11], carbohydrate (by different), and total calories.

Table 1. Formula of cookies

| Formula | Mocaf | Egg-tempe flour | Green bean flour | Catfish flour | Butter | Sugar | York | Mocha pasta |
|---------|-------|-----------------|------------------|---------------|--------|-------|------|-------------|
| F1 | 68 g | 5 g | 12 g | 15 g | 50 g | 60 g | 32 g | 9 g |
| F2 | 63 g | 5 g | 12 g | 20 g | 50 g | 60 g | 32 g | 9 g |
| F3 | 58 g | 5 g | 12 g | 25 g | 50 g | 60 g | 32 g | 9 g |

Shelf life was hypothesized using ASLT method based on critical water content. The stages include: (1) determination of initial moisture content based on Thermogravimetry method (M_i)[11]; (2) determination of critical moisture content (M_c); (3) determination of equilibrium moisture content (M_e); (4) determination of Packaging Permeability on Water Vapour (ASTM E96/E96M-10); and (5) determination of shelf life (t) [9].

$$\ln \left[\frac{M_e - M_i}{M_e - M_c} \right] = (k / x) \left[\frac{A}{W_s} \right] \left[\frac{P_o}{b} \right] \Theta \quad (1)$$

Where :

M_e : equilibrium moisture content (g H_2O / 100 g solid)

M_i : initial moisture content (g H_2O / 100 g solid)

M_c : critical moisture content (g H_2O / 100 g solid)

k/x : moisture permeability through packaging material (g H_2O (m^2 day mm Hg) $^{-1}$)

A : Packaging area (m^2)

W_s : weight of dry food solids (g)

P_o : water vapor pressure at the storage temperature (mmHg)

b : slope of the linearized isotherm portion (i.e., from M_i to M_c)

3. Results and Discussion

3.1. Preference Test

Results of cookies preference test are shown in Table 2. The parameter of the most preferred color, aroma, texture and overall preference is formula F2. However, Formula F1 has a preference score

equal to parameters of overall aroma and preference. Formula with the lowest preference rate is Formula F3. Preferred overall Formula is Formula F1 and F2. However, if seen from some of the other parameters, chosen Formula is Formula F2. Thus, furthermore, the Formula F2 was tested chemically, and shelf life was specified.

Table 2. Preference Scores of Cookies

| Formula Cookies | Parameter | | | | |
|--------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| | Color | Aroma | Taste | Texture | Overall |
| F1 | 3.64 ^a | 3.64 ^a | 3.80 ^a | 3.84 ^a | 3.92 ^b |
| F2 | 3.72 ^a | 3.64 ^a | 3.80 ^a | 4.00 ^a | 3.92 ^b |
| F3 | 3.32 ^a | 3.28 ^a | 3.36 ^a | 3.56 ^a | 3.40 ^a |

Where: Different notations in the same column show real differences in α 5%

3.2. Chemical Analysis

Results of selected Formula (F2) analysis indicate that water content 6.01%; ash content 1.77%; protein content 12.06%; fat content 20.99%, carbohydrate content, 59.17%; energy 511.56 kcal/100 g. The total energy of Formula F2 meets minimal quality requirement of total calories with SNI 01-2992, minimal 400 kcal/100 g [12].

3.3. Shelf life prediction

3.3.1. Initial moisture Content (Mi)

The initial moisture content of cookies was 5.02%. The low water content of cookies is caused by baking process at high temperature (108 °C) making sucrose re-crystallization discharge water steam [13].

3.3.2. Critical moisture content (Mc)

The crunchiness of cookies is stated as a critical parameter of damage to cookies because it is most easily impaired. This is caused by the absorption of moisture from the environment which can increase the moisture content of the product. Critical moisture content is expressed as water content where the crispness of the product can no longer be accepted organoleptically by consumers [6]. Scores of water content and organoleptic score during storage are shown in Figure 1.

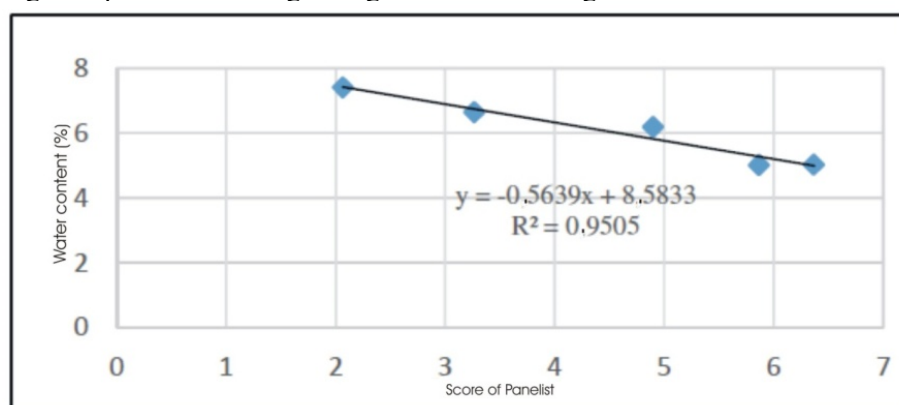


Figure 1. Correlation between water content and an organoleptic score of cookies

The graph shows that increasing the water content of the cookies panelists score is getting smaller, meaning that the panelists' preference has decreased. This shows a decrease in quality due to the increasingly soft texture due to absorption of moisture from the environment [6]. The linear equation obtained is $Y = -0.5639X + 8.5833$ with R^2 of 0.95. From these linear equations can be determined the value of critical water content by plotting a score of 3 on the x value so that the critical water content of cookies is protein and energy source of 6.8916%. The initial moisture content of this product

determines critical moisture content and will affect the material adsorption process to the environment to achieve equilibrium conditions [14].

3.3.3. Equilibrium moisture content (M_e)

The equilibrium moisture content of various levels of a_w based on the results of the research is shown in Table 3. The higher value of water activity (a_w), value of balanced water content of cookies is also higher. The difference in the level of a_w value from various salts is affected by the reaction between water and ionic bonds between the atoms that make up the salt. The ionic bonds between atoms the greater the activity of the salt water. Water activity is expressed as the chemical potential of water whose value varies from 0 to 1. If the water activity value is equal to 0 then the water molecule concerned does not carry out any activity in the chemical process, or the material is dry. If the water activity value is equal to 1 then the water potential in the chemical process at maximum conditions or material is pure water [6].

Table 3. Equilibrium moisture content Cookies at Various RH Storage

| Salt | Water activities (a_w) | Balanced Water Content (M_e) (%) |
|-----------------------------------|----------------------------|--------------------------------------|
| LiCl | 0.112 | 2.3197 |
| KCH ₃ CO ₂ | 0.222 | 3.7545 |
| MgCl ₂ | 0.325 | 4.9368 |
| K ₂ CO ₃ | 0.437 | 6.6954 |
| Mg(NO ₃) ₂ | 0.519 | 8.2006 |
| KI | 0.682 | 12.7303 |
| NaCl | 0.752 | 14.9064 |
| KCl | 0.838 | 22.7530 |
| K ₂ SO ₄ | 0.967 | 41.4999 |
| LiCl | 0.112 | 2.3197 |
| KCH ₃ CO ₂ | 0.222 | 3.7545 |

Table 3 shows the level of water absorbed by cookies to achieve equilibrium is strongly influenced by relative humidity (RH). The higher the RH, the greater the amount of water vapor absorbed by the material to achieve balance [15]. This equilibrium moisture content is then plotted with relative humidity or water activity (a_w) and forms a curve called the moist sorption isotherm curve (ISL). Moist sorption isotherm curves relationship between (a_w) and balanced water content are shown in Figure 2.

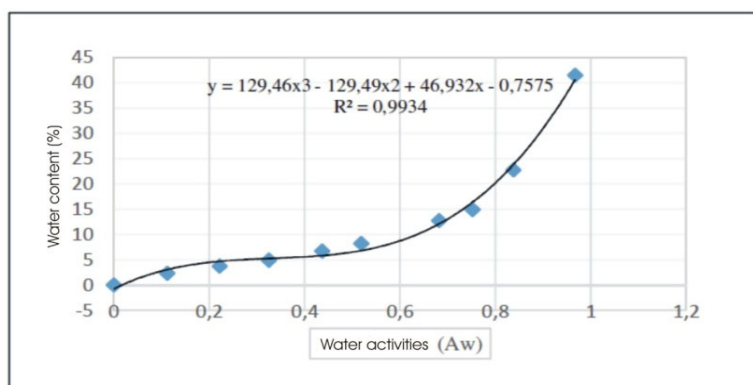


Figure 2. ISL Curve of Cookies

The relationship between water activity (a_w) and balanced water content of cookies in Figure 2 is made using the degree three polynomial equation. The constant equation produced $A = 129.46$; $B = -129.49$; $C = 46.93$; $D = -0.75$ with a value of $R^2 = 0.99$. Thus the equation of Isotherm Sorpsi Moist

Cookies is a source of protein and energy as follows: $Y = 129.46x^3 - 129.49x^2 + 46.93x - 0.75$, (y = equilibrium moisture content (%); $x = a_w$).

The general form of the GAB equation is used to calculate the a_w value of the initial moisture content (M_i) and critical water content (M_c) of cookies. The initial water content, critical water content, and water activity are plotted into the GAB equation to obtain an equilibrium moisture content (M_e). Data on determining the GAB curve are presented in Table 4.

Table 4. Data of GAB Equation Specification of Cookies

| Salt | A_w | a_w Salt/Balanced Water Content |
|-----------------------------------|-------|-----------------------------------|
| LiCl | 0.112 | 0.0483 |
| KCH ₃ CO ₂ | 0.222 | 0.0591 |
| MgCl ₂ | 0.325 | 0.0657 |
| K ₂ CO ₃ | 0.437 | 0.0653 |
| Mg(NO ₂) ₂ | 0.519 | 0.0633 |
| KI | 0.682 | 0.0520 |
| NaCl | 0.752 | 0.0463 |
| KCl | 0.838 | 0.0368 |
| K ₂ SO ₄ | 0.967 | 0.0233 |

Based on Table 4, it is furthermore plotted in GAB curve using degree 2 polynomial Equation. Water activity (a_w) as x-axis and comparison of a_w with equilibrium water content as the y-axis. The GAB curve formed can be seen in Figure 3.

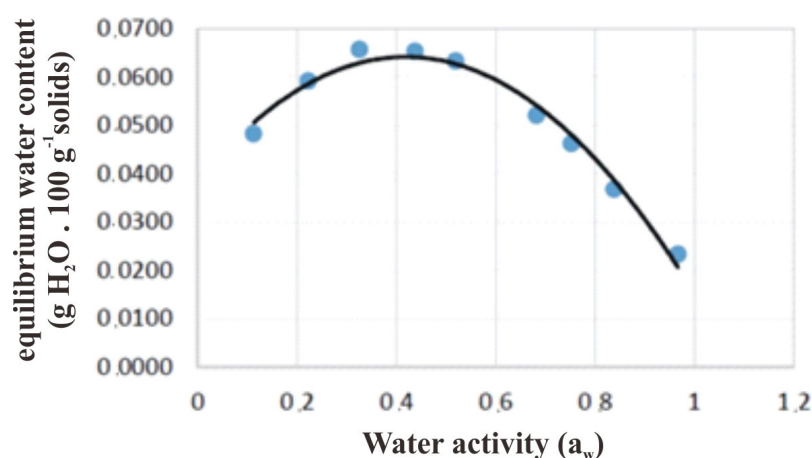


Figure 3. GAB Equation Curve of Cookies

The equation for GAB cookies is shown in Figure 3, $Y = -0.1502x^2 + 0.1286x + 0.0374$ with $R^2 = 0.984$. The K , C and M_o values obtained based on the Bizot [16] procedure in this study were 0.92; 5.73; and 5.06. C and K values show monolayer and multilayer adsorption energies. The value of C is adsorption energy associated with the water binding energy in the monolayer layer is quite low. The low energy of the adsorption is related to the type of bond involved in the binding of water. The greater the value of C , the stronger the bond between water molecule of monolayer and material surface layer is stronger in bonding [17].

The sigmoid sorption type isotherm is well described if the values are $0.24 < K \leq 1$ and $5.67 \leq C \leq \infty$. While value of the monolayer water content is very necessary to determine the storage conditions and control of the damage reactions that may occur and determine the physical and chemical conditions of the dry material [18]. The water content of this monolayer layer is primarily bounded

water, which cannot function as a solvent. The primarily bonded water is part of the solid because the water is absorbed on the active side of the polar solids [19]. The general form of GAB cookies is:

$$M = \frac{26,738 a_w}{(1-0,9212 a_w)(1-0,9212 a_w+5,2809 a_w)} \quad (2)$$

Values of M_i , M_c , and a_w can be understood by the general form of GAB Equation. Values of M_i , M_c and a_w are shown in Table 5.

Table 5. Values of a_w of initial material (M_i), critical material (M_c), and balance (M_e)

| Parameter | | Value |
|--|------------------------|-----------------------|
| Determination a_w of initial material | M_i | 5.0231 |
| | $a_w M_i$ | 0.3168 |
| Determination a_w of Critical moisture content (M_c) | M_c | 6.8916 |
| | $a_w M_c$ | 0.557 |
| Determination of M_e | Regressive Equation of | $Y = 7.780x + 2.5586$ |
| | $a_w M_e$ | 0.75 |
| | M_e | 8.3934 |

From the Equation, value of a_w is plotted so that value of *Equilibrium moisture content* (M_e) 8.3934%.

3.3.4. Moisture Permeability

Dry food such as cookies is hygroscopic so that these need packaging process to prevent water steam from entering into the product. Data for packaging permeability test results for water steam are shown in Table 6.

Table 6. moisture permeability through packaging material & Slope

| Packaging | Slope | k/x (gH ₂ O/day.m ² .mmHg) | Mean |
|---------------|---------|---|-------|
| Aluminum Foil | 0.00027 | 0.0039 | 0.004 |
| | 0.00024 | 0.0035 | |
| | 0.00026 | 0.0038 | |
| Metalizing | 0.00042 | 0.0061 | 0.006 |
| | 0.00045 | 0.0066 | |
| | 0.00044 | 0.0064 | |
| PE | 0.00349 | 0.0508 | 0.050 |
| | 0.00315 | 0.0459 | |
| | 0.00364 | 0.0531 | |

Based on Table 6, it can be understood that value of slope is directly proportional to packaging permeability. The higher value of packaging permeability, the water steam will be easier to penetrate the packaging, so that it will be easier to achieve critical water content and shorten shelf life [19]. Packaging permeability is found good and quality if it has low value. The lower value of packaging permeability, the shelf life is longer [6].

3.3.5. Shelf Life

Based on Equation of GAB curve (Figure 3), value of Balanced Water Content with RH = 75% (RH of place in which cookies is stored) is 8.3934%. Slope (b) or slope of the isothermal curve is obtained by plotting data of initial water content and critical water content in linear Equation. Linear Equation passing M_e and M_c is shown in Figure 4, where value of slope is 7.7798.

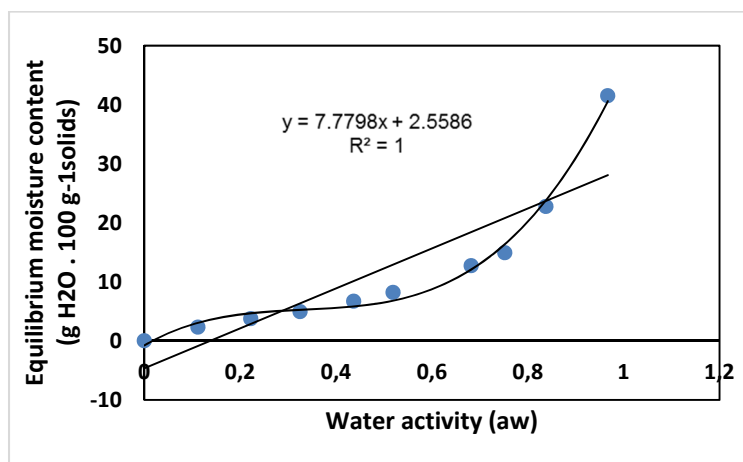


Figure 4. Linear Equation passing initial water content (Me) and critical water content (Mc)

The measure of packaging used in calculating the shelf life is 10 cm x 12 cm where packaging area is 0.024^2 in both sides. This measure is adapted to packaging used to package cookies in markets. Available data are plotted in Labuza Equation, so that hypothesis of shelf life of cookies can be seen in Table 7.

Table 7. Results of Shelf life Hypothesis of Cookies

| Pack-aging | Mi | Mc | Me | k/x | A (m ²) | Ws (gram) | Po (mmHg) | Slope ISL | Shelf life | |
|------------|--------|--------|--------|-------|---------------------|-----------|-----------|-----------|------------|-------|
| | | | | | | | | | Day | Month |
| A | 5.0231 | 6.8916 | 8.3934 | 0.004 | 0.024 | 23.80445 | 28.349 | 0.0778 | 588,05 | 19,6 |
| B | 5.0231 | 6.8916 | 8.3934 | 0.006 | 0.024 | 23.80445 | 28.349 | 0.0778 | 366.70 | 12,2 |
| C | 5.0231 | 6.8916 | 8.3934 | 0.050 | 0.024 | 23.80445 | 28.349 | 0.0 | 44,05 | 1.5 |

A : Aluminum foil; B: Metalizing; C: polyethylene.

Shelf lives of cookies use polyethylene packaging, metalizing, and aluminum foil are, respectively, 1.5 months; 12 months; and 19 months. Based on Table 7, it can be understood that the higher packaging permeability rate, shelf life is shorter. It is concordant to the theory of shelf life specification principle suggested by [19] indicating that the higher packaging permeability, the water steam is easier to penetrate the packaging so that it will be easier to achieve critical water content and shorten shelf life.

Packaging area and solid weight of packaging affect the shelf life of the product. The wider area of packaging, the entering water steam is wider to distribute to packaging and prevent achievement of critical water content, and total solidity is less in packaging so that shelf life will be shorter. It is caused by water steam entering into the packaging is distributed widely and accelerates achievement of critical water content [19]. This research used the packaging area of 0.024 m^2 and same solid weight, 23.8 g so that effect on shelf life is not seen clearly.

4. Conclusions

This research indicates that cookies with the best formula are formula F2 (mocaf = 63%; egg-tempe flour, 5%; green bean flour, 12%; catfish flour, 20%), contain 12.06% of protein, and energy of 511.56 kcal/100 g. The critical water content of cookies is 6.8916%. Values of polyethylene packaging permeability, metalizing, and aluminum foil are, respectively, $0.05 \text{ gH}_2\text{O/day.m}^2.\text{mmHg}$, $0.006 \text{ gH}_2\text{O/day.m}^2.\text{mmHg}$, and $0.006 \text{ gH}_2\text{O/day.m}^2.\text{mmHg}$. The three packagings are able to remain qualities respectively during 1.5 months, 12 months, and 19 months.

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