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ACCELERATED SHELF LIFE TEST (ASLT) METHOD WITH ARRHENIUS APPROACH FOR SHELF LIFE ESTIMATION OF SUGAR PALM FRUIT JAM WITH ADDITION OF ASIAN MELASTOME (*Melastoma malabathricum*, L.) ON JAR PACKAGING AND POUCH

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Abstract. Base on previous studies showing that sugar palm fruit can be made as jam with the addition of Asian melastome fruit as a natural coloring. This sugar palm fruit jam product is packaged with jar and pouch packaging. shelf Life information for this product is unknown. The purpose of this study is to determine the rate of change in quality, critical parameters, and estimate the shelf life of sugar palm fruit jam with Arrhenius Model which is simulated at three storage temperature conditions (28°C, 38°C, and 48°C). Parameters observed during the storage process are water content, water activity (a_w), pH, color (L), anthocyanin, and antioxidant activity (IC_{50}). The results showed that the smallest activation energy value was used to determine product shelf life, it is color parameters (L) (first order reaction) with linear regression $y = -4715.x + 9,787$ on jar bottle packaging and $y = -2021.x + 1,387$ on packaging pouch. The age of storing sugar palm fruit jam with jar bottles is 93 days at 28°C and on the packaging pouch for 10 days at 28°C.

Keywords: Accelerated Shelf Life Test (ASLT), arrhenius, sugar palm jam, self life, asian melastome

1. Introduction

Today the development of products with appropriate technology that comes from sugar palm fruit is beginning to increase, which one is jam products. Making jam using raw materials sugar palm fruit has deficiencies in terms of appearance, because the color of the jam produced is white that is less attractive. For this reason, the addition of natural dyes is one of them by using asian melastome fruit. Asian melastome fruit can be used as a natural coloring agent because it has anthocyanin pigments that can give purplish color to the product. Sugar palm fruit jam has a high water content, this will greatly affect the storage process and packaging selection for sugar palm fruit jam products (Sayuti et al., 2017). Packaging is important in extending the storage capacity of agricultural produce. Placing a material or product can make it easy to facilitate storage, transportation and distribution to the buyer's community is a function rather than packaging.

Sugar palm fruit is obtained from boiling endosperm of young sugar palm fruit seeds that have various nutrients that are beneficial for healthy. In addition, sugar palm

fruit also contains vitamins A, B, and C, and fiber contains which can help the digestive tract work, 52.9% carbohydrates, especially galactomannan which has analgesic or pain relief effects so that it can reduce pain in arthritis (Hidayat& Napitupulu, 2015). Galactomannan is a carbohydrate reserve that functions as a regulator of the amount of water in seeds during germination. Galactomannan is also a thickener and emulsion stabilizer that is good and can reduce the risk of toxic entry if used as a pharmaceutical ingredient and food industry (Brooks et al., 2005). Some research have reported that anthocyanin is also attractive in its biological activity, namely antioxidant activity (Kong et al., 2003), anti-inflammatory, anticarcinogenic (Katsube et al., 2003), antidiabetic (Jayaprakasha et al., 2006), neuroprotective (Galli et al., 2002), antimutagenic, antitumor, and hepatoprotective (Kong et al., 2003). Anthocyanin can also reduce the risk of coronary heart disease through inhibition of platelet aggregation (Ghiselli et al., 2002) and the inhibition of LDL (low density lipoprotein) lipoprotein oxidation (Heinonen et al., 1998).

Food product is produced must be known for the shelf life it can be known about expiration date, that can be safe food products for consumption. During storage, physical and chemical changes will occur from the product, it can reduce the quality and taste of the products produced (Arpah, 2001). In estimating shelf life can use several methods, in the sugar palm fruit jam product, the shelf life determination using the Accelerated Shelf Life Test (ASLT) method in Arrhenius Model. Where this method is a method that speeds up shelf life by increasing the temperature measured (Arpah. 1998).

The shelf life of food products is vulnerable to time between production and consumption where the product is in good condition and accepted by the consumer based on the characteristics of appearance, taste, aroma, and nutritional value. Shelf life is obtained from the lowest activation energy value of the critical value of food quality. The principle of the ASLT method with the Arrhenius model is to accelerate the physical and chemical damage of food products by increasing temperature, then shelf life can be determined by mathematical calculations.

2. Materials and Methods

Tools and Materials

The materials used to make sugar palm fruit jam include fresh sugar palm fruit which is indicated by white flesh, clean and soft texture. And added sugar, asian melastome fruit and citric acid. The material used in estimating shelf life is the sugar

palmfruit jam product with the addition of asian melastome fruit which is packaged in pouch packaging made from Polypropylene (PP) plastic spoutpack (cap seal) type with a thickness of 0.08 mm and 150 gram size, then packaging glass jar bottles with handles measuring 120 grams. ingredients for analysis are meades, pH 4 buffer solution and pH 7, saturated ammonium oxalate solution, indicator methyl red, acetic acid, HCl, AgNO₃, H₂SO₄, KMNO₄ 0.01 N, NaOH, K₂SO₄ 10%, 95% alcohol, CaCl₂ 1 N , potassium chloride buffer solution (pH 1.0), sodium acetate buffer solution (pH 4.5), methanol, DPPH, and other analytical ingredients. The tool used to condition the storage is an incubator. The tools used in the analysis are a set of glassware, spatulas, analytical scales, blenders, filters, rattles, desiccators, water baths, pH meters, UV-VIS spectrophotometers, thermometers, pipet, a_w meters, funnels, and other supporting tools.

Research Methods

The process of making sugar palm fruit jam is as follows:

- a. Fresh sugar palmfruit are washed with clean water,
- b. Sugar palm fruit is blansing for \pm 15 minutes,
- c. Sugar palm fruit is crushed using a blender to be porridge,
- d. The porridge from sugar palm fruit is weighed 45 grams, 55 grams of sugar and 0.3 grams of citric acid,
- e. The sugar palm fruit porridge and sugar are included in the mixing bowl equipped with a stirrer, at a temperature of 40 oC, stirred until homogeneous,
- f. Citric acid and asia melastome fruit which have been cleaned are put into a mixture of sugar palm fruit porridge and sugar at a temperature of 40 oC,
- g. Cooking is continued for 10-20 minutes. During cooking, stirring is carried out continuously. Stirring should not be too fast because it will cause bubbles that can damage the texture and appearance of the final jam,
- h. The spoon test is performed to see the formation of jam. Spoon test is done by taking a little mixture with the tip of the spoon, left to cool for a while then the spoon is tilted, if it does not fall immediately the cooking process can be stopped.

The product of sugar palm fruitjam produced is packaged in 120 gram jar and 150 gram pouch. The sugar palm fruit jam in bottles jar and pouch packaging that have just been produced are grouped into three and each stored at 28 °C, 38 °C and 48°C for one month (30 days). Observations were made for every 0, 10, 20, 30 day curry for each storage temperature. The characteristic parameters of the kolang - kaling jam products

tested were water content, a_w value, pH, color (L), IC_{50} and anthocyanin. Observations made on critical parameters of color value (L) with hunter LAB tools.

Shelf Life Estimated

Estimation of shelf life using the Accelerated Shelf Life Testing (ASLT) method that uses the influence of temperature to accelerate damage with the Arrhenius approach (Herawati, 2008). Shelf life estimation steps using the ASLT method with the Arrhenius approach as follows:

Data from product analysis on time are plotted and the linear regression equation is calculated. Then three regression equations are obtained for the three conditions of product storage temperature using $Y = a + bx$, where:

Y = the characteristic value of jam,

X = storage time (days),

a = value of characteristic of juice at the beginning of storage,

b = rate of change in characteristic value (value b equals value of k).

From each of these equations, the slope value (b) which is the reaction rate constant changes the product characteristics or the rate of degradation (k). The constant value of quality degradation (k) per day is obtained from the slope of the regression equation of the two graphs. After obtaining a value of k, then look for the value of ln for each storage temperature. Arrhenius plots were then made, with the x axis declaring $1/T$ (K⁻¹) and the y axis indicating ln k at each storage temperature used (28°C, 38°C and 48°C). The value of k is the gradient of linear regression obtained from the three storage temperatures. From the linear regression obtained on the Arrhenius curve it can be predicted the shelf life of the product using the formula:

$$k = k_0 e^{-(E_a/RT)}$$

information :

k = quality change constant

k_0 = constant (not dependent on temperature)

E_a = activation energy

T = absolute temperature (oK)

R = gas constant (1.986 cal / mol oK or 8.314 J / mol. °K)

By changing the above equation to:

$$\ln k = \ln k_0 + (-E_a/R)1/T$$

k_0 is the constant of product quality degradation which does not depend on

temperature, while k is the quality reduction constant of one of the temperature conditions used (28 °C, 38 °C and 48°C) and Ea / R is the gradient obtained from the Arrhenius plot. With calculations using this formula, a value of k₀ will be obtained. The shelf life according to the order of reaction one is obtained by the formula:

$$t = \frac{\ln A_0 - \ln A_t}{K_0}$$

information :

t = prediction of shelf life (days)

A₀ = initial quality value (Parameter initial value)

A_t = value of product quality remaining after time t

k₀ = constant

From the formula above can be predicted shelf life in days or months. If the reaction follows a zero reaction order, then the shelf life can be obtained using the formula:

$$t = \frac{A_0 - A_t}{K_0}$$

information :

t = prediction of shelf life (days)

A₀ = initial quality value (initial moisture content)

A_t = value of product quality remaining after time t

k₀ = constant

From the formula above can be predicted shelf life in days or months.

3. Results and Discussion

Jam is stored in an incubator at temperatures of 28°C, 38°C and 48°C, in two types of packaging, plastic pouch and glass jar. Then chemical analysis was carried out as before storage (on day-0). The results of the analysis are used as quality characteristics of the final kolang jam kaling (A_t) can be seen in Table 1.

Table 1. Quality characteristics of sugar palm fruit jam before storage (A₀)

Parameter	Analysis Results
	(\bar{X})
Moisture content (%)	32,480
Color	
- L*	7,743
- a*	1,556
- b*	4,815
- ⁰ Hue	17,909
a _w	0,747
Ph	3,375

Antosianin (mg/l)	14,730
Aktivitas Antioksidan (IC ₅₀) (mg/ml)	9,77

Table 2. Quality characteristics of sugar palm fruit jam after storage (At) during 30 days with bottle jar packaging and Pouch packaging

Parameter	Analysis Results with bottle jar packaging	Analysis Results with pouch packaging
	(\bar{X})	(\bar{X})
Moisture content (%)	34,720	37,827
Color		
- L*	10,055	9,423
- a*	5,955	4,260
- b*	0,825	0,453
⁰ Hue	82,113	83,930
a _w	0,758	0,758
pH	4,450	4,223
Antosianin (mg/l)	1.336	0.5845
Aktivitas Antioksidan (IC ₅₀) (mg/ml)	47,348	52,276

Basic Reaction Kinetics To Estimate Decreasing Quality of Sugar Palm Fruit Jam

Basic reaction kinetics are calculated from each product stored at 28⁰C, 38⁰C, and 48⁰C through chemical analysis including water content and color (L value).

Table 3. Value of changes in moisture content during storage on jar packaging and packaging pouch

Days	Moisture Content (%)					
	Jar Packaging			Pouch Packaging		
	28 ⁰ C	38 ⁰ C	48 ⁰ C	28 ⁰ C	38 ⁰ C	48 ⁰ C
0	32,48	32,48	32,48	32,480	32,480	32,480
10	31,915	32,215	34,104	33,590	34,242	34,940
20	32,450	33,431	34,671	33,627	34,988	35,005
30	32,741	33,097	34,720	33,827	36,354	37,827

Table 4. Value of changes in color during storage on jar packaging and packaging pouch

Days	Color (L)					
	Jar Packaging			Pouch Packaging		
	28 ⁰ C	38 ⁰ C	48 ⁰ C	28 ⁰ C	38 ⁰ C	48 ⁰ C
0	7,743	7,743	7,743	7,743	7,743	7,743
10	7,865	7,855	7,76	7,787	7,717	8,32
20	8,16	8,545	8,54	9,887	8,757	8,41
30	8,57	8,725	10,055	9,65	8,61	9,423

Based on Figure 5 above, a straight line equation and a slope value at each storage temperature are obtained. The selection of order kinetics of the reaction for decreasing water content is done by comparing the value of the correlation coefficient (R²) of each linear regression equation at the same temperature of the zero order reaction (A plotted

against time) and first order reaction (ln A plotted against time). The reaction order with a greater R^2 value is the reaction order used (Labuza, 1982 in Wasono and Yuwono, 2014). The choice of order for changes in water content on jar bottle packaging and packaging pouch can be seen in Table 5 and 6.

Table 5. The reaction equation for the moisture content parameters in the zero order jar bottle and the first order in the sugar palm fruit jam

Temperature (°K)	Reaction Equation		R ²	
	Ordo Zero	Ordo One	Ordo Zero	Ordo One
301 (28°C)	$y = 0,013x + 32,19$	$y = 0,000x + 3,471$	0,241	0,239
311 (38°C)	$y = 0,030x + 32,34$	$y = 0,000x + 3,476$	0,505	0,050
321 (48°C)	$y = 0,072x + 32,9$	$y = 0,002x + 3,493$	0,807	0,803

Table 6. The reaction equation for the moisture content parameters in the zero order pouch package and the first order in the sugar palm fruit jam

Temperature (°K)	Reaction Equation		R ²	
	Ordo Zero	Ordo One	Ordo Nol	Ordo Satu
301 (28°C)	$y = 0,040x + 32,76$	$y = 0,001x + 3,489$	0,745	0,743
311 (38°C)	$y = 0,123x + 32,66$	$y = 0,003x + 3,486$	0,977	0,974
321 (48°C)	$y = 0,161x + 32,64$	$y = 0,004x + 3,486$	0,905	0,906

From the Table above, the **sugar palm fruit jam product with jar and pouch** bottles can be seen that in both packages has zero order correlation coefficient greater than the first order correlation coefficient (zero order $R^2 > R^2$ first order), then the rate of decreasing water content sugar palm fruit jam on jar bottles and pouch packs follows a zero order reaction.

The slope value of the equation is the value of $-E / R$ from the Arrhenius equation, so that it can be obtained the activation energy of kang-kaling jam on each packaging as follows:

Table 7. Energy activation of sugar palm fruit jam on each packaging

Packaging Type	-E/R	R	E
Jar	-8264	1,986	4161,128
Pouch	-6769	1,986	3408,359

Furthermore, the selection of order of changes in color value (L) can be seen in Table 8.

Table 8. The reaction equation for the color parameter (L value) on the zero-order jar bottle and the first order on the sugar palm fruit jam

Temperature (°K)	Persamaan reaksi		R ²	
	Ordo Zero	Ordo One	Ordo Zero	Ordo One

301 (28°C)	$y = 0,027x + 7,668$	$y = 0,003x + 2,038$	0,948	0,953
311 (38°C)	$y = 0,036x + 7,671$	$y = 0,004x + 2,038$	0,916	0,916
321 (48°C)	$y = 0,077x + 7,367$	$y = 0,008x + 2,005$	0,841	0,855

Table 9. The reaction equation for the color parameter (Value L) in the zero order pouch package and the first order in the sugar palm fruit jam

Temperature (°K)	Reaction equation		R ²	
	Ordo Zero	Ordo One	Ordo Zero	Ordo One
301 (28°C)	$y = 0,078x + 7,593$	$y = 0,004x + 2,036$	0,756	0,723
311 (38°C)	$y = 0,036x + 7,660$	$y = 0,009x + 2,029$	0,720	0,763
321 (48°C)	$y = 0,051x + 7,704$	$y = 0,006x + 2,044$	0,899	0,909

The slope value of the equation is the value of $-E / R$ from the Arrhenius equation, it can be obtained the activation energy of sugar palm fruit jam on each packaging as follows:

Table 10. Energy activation of sugar palm fruit jam in each packaging

Packaging Type	-E/R	R	E
<i>Jar</i>	-4715	1,986	2374,119
<i>Pouch</i>	-2021	1,986	1017,623

Estimating Self Life of Sugar Palm Fruit Jam

The activation energy value (Ea) is obtained from the slope of the equation of each parameter, then one parameter is selected which most influences the decrease in the quality of sugar palm fruit jam during storage in various temperature variations, the parameter which has the lowest activation energy (Ea) value. The following table is the activation energy comparison for each analysis parameter obtained.

Table 11. Activation energy value for each parameter of sugar palm fruit jam on a jar bottle

No	Analysis Parameters	Activation Energy (kal / mol)
1	Moisture content	4161,128
2	Color (L)	2374,119

Table 12. Activating energy values for each parameter of sugar palm fruit jam on pouch packaging

No	Analysis Parameters	Activation Energy (kal / mol)
1	Moisture content	3408,359
2	Color (L)	1017,623

Based on Table 11 and Table 12 which has the lowest activation energy (Ea) which is the color parameter (L value). The following is a linear regression equation for the

graph of the relationship $1/T$ with $\ln k$ in the color parameter (value L) of sugar palm fruit jam on the jar bottle packaging and packaging pouch.

Bottle jar packaging

The linear regression equation for the graph of the relationship $1/T$ with $\ln k$ in the color parameter (L value) of sugar palm fruit jam is $y = -4715.x + 9,787$ and $R^2 = 0.937$.

Who has activation energy of:

$$-E/R = -4715K$$

$$R = 1,986 \text{ kal/mol K}$$

$$E = 2374,119 \text{ kal/mol}$$

The intercept value is the value $\ln k_0$ of the Arrhenius equation, so:

$$\ln k_0 = 9,787$$

$$k_0 = 17800,82$$

Based on the E/R and k_0 values that have been obtained, the Arrhenius equation can be arranged as follows:

$$K = k_0 \cdot e^{-E/RT}$$

$$K = 17800,82 \cdot e^{-4715(1/T)}$$

The rate of increase in color (L value) on the sugar palm fruit jam based on each temperature can be calculated after obtaining the Arrhenius equation, as follows:

$$28^\circ\text{C or } 301 \text{ K } K = 17800,82 \cdot e^{-4715(1/T)}$$

$$K = 17800,82 \cdot e^{-4715(1/301)}$$

$$K = 0,002802$$

$$38^\circ\text{C or } 311 \text{ K } K = 17800,82 \cdot e^{-4715(1/T)}$$

$$K = 17800,82 \cdot e^{-4715(1/311)}$$

$$K = 0,004637$$

$$48^\circ\text{C or } 321 \text{ K } K = 17800,82 \cdot e^{-4715(1/T)}$$

$$K = 17800,82 \cdot e^{-4715(1/321)}$$

$$K = 0,007436$$

Sugar palm fruit jam shelf life in jar packaging at each temperature:

Temperature		K	Self life (Day)
$^{\circ}\text{K}$	$^{\circ}\text{C}$		
301	28	0,002802	93,24797 ~ 93
311	38	0,004637	56,34695 ~ 56
321	48	0,007436	35,13728 ~ 35

Pouch packaging

The linear regression equation for the graph of the relationship $1/T$ with $\ln k$ in the

color parameter (L value) kaling jam is $y = -2021.x + 1.387$ and $R^2 = 0.266$. Who has activation energy of:

$$\begin{aligned} -E/R &= -2021K \\ R &= 1,986 \text{ kal/mol K} \\ E &= 1017,623 \text{ kal/mol} \end{aligned}$$

The intercept value is the value $\ln k_0$ of the Arrhenius equation, so:

$$\begin{aligned} \ln k_0 &= 1,387 \\ k_0 &= 4,002824 \end{aligned}$$

Based on the E / R and k_0 values that have been obtained, the Arrhenius equation can be arranged as follows:

$$\begin{aligned} K &= k_0.e^{-E/RT} \\ K &= 4,002824. e^{-2021(1/T)} \end{aligned}$$

The rate of increase in color (L value) on the sugar palm fruit jam based on each temperature can be calculated after obtaining the Arrhenius equation, as follows:

$$\begin{aligned} 28^\circ\text{C or } 301 \text{ } ^\circ\text{K} \quad K &= 4,002824. e^{-2021(1/T)} \\ &= 4,002824. e^{-2021(1/301)} \\ &= 0.004857 \\ 38^\circ\text{C or } 311 \text{ } ^\circ\text{K} \quad K &= 4,002824. e^{-2021(1/T)} \\ &= 4,002824. e^{-2021(1/311)} \\ &= 0.006028 \\ 48^\circ\text{C or } 321 \text{ } ^\circ\text{K} \quad K &= 4,002824. e^{-2021(1/T)} \\ &= 4,002824. e^{-2021(1/321)} \\ &= 0.007380 \end{aligned}$$

Sugar palm fruit jam shelf life in pouch packaging at each temperature:

Temperature		K	Self life (Day)
$^\circ\text{K}$	$^\circ\text{C}$		
301	28	0.004857	40,42913 ~ 40
311	38	0.006028	32,57536 ~ 33
321	48	0.007380	26,60763 ~ 27

4. Conclusions

The conclusions of the initial quality characteristics of sugar palm fruit jam products with the addition of asian melastome fruit tested in this study had a moisture content of 32.480%, water activity (a_w) 0.747, pH 3.375, color value (L) 7.743, anthocyanin 14.730 ml/L and antioxidant activity (IC_{50}) of 9.77 mg/ml. The results of the

final quality characteristics of sugar palm fruit jam products with the addition of asian melastome fruit tested in this study had a moisture content of 34.720% in jar packaging and 37.827% in pouch packaging, water activity (a_w) on both packages of 0.758, pH value on jar packaging 4,450 and on pouch 4,223, color value (L) on jar 10,055 and on pouch 9,423, anthocyanin on jar 1,336 mg/l and on pouch 0,585 mg/l, and antioxidant activity (IC_{50}) at 47,348 mg/ml on jar packaging and on the pouch packaging 52,276 mg/ml. The initial quality characteristics to the final quality characteristics of the sugar palm jam showed that the temperature greatly affected the rate of damage of the sugar palm fruit jam products. The higher the storage temperature, the faster the damage to the product will be.

The calculation results of sugar palm fruit jam shelf life are based on the color parameter (L value), because it has a lower activation energy value than using the moisture content parameter, which is 2374,119 kal/mol in jar bottle packaging and 1017,623 cal/mole on pouch packaging. The shelf life of sugar palm fruit jam obtained in packaging using jar bottles for storage of room temperature 28 °C is 93 days and in the use of pouch packaging is 40 days, the shelf life of sugar palm fruit jam at 38 °C which is packed with jar bottles is 56 days and on products packed with pouches of 33 days, and shelf life of sugar palm fruit jam for storage at 48 °C in jar packaging is 35 days and on pouch packaging is 27 days.

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