

## Kinetics of Changes in Free Fatty Acids in Cassava Crackers (Kecimpring) Enriched with Catfish and Gotu Kola (*Centella asiatica*) Leaves During Storage

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### ABSTRACT

Cassava crackers, also known as kecingpring, are a traditional Indonesian snack that is low in protein but high in carbohydrates. Researchers have explored fortifying these crackers with catfish to enhance their nutritional value and increase their protein content. However, the high fat content in catfish can increase overall lipid levels in the crackers, making them more susceptible to oxidation during storage. Using the Arrhenius model, this study aimed to evaluate the kinetics of free fatty acid (FFA) formation in fortified cassava crackers. The enriched product, which included catfish and *Centella asiatica* leaves, was stored at temperatures of 25°C, 35°C, and 45°C for 28 days in polypropylene packaging that is 0.4 mm thick. FFA levels were measured at 7-day intervals. The results revealed an activation energy ( $E_a$ ) of 54.01 kJ/mol, indicating that higher storage temperatures accelerate lipid hydrolysis and FFA accumulation. These findings suggested that controlling the storage temperature was crucial to maintaining the quality and stability of fortified cassava crackers.



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## 1. Introduction

Cassava kecipring crackers is a traditional Indonesian snack popular among various social groups (Lis Rostini, 2016). However, it has relatively low levels of protein. One potential strategy to enhance the nutritional value of cassava crackers kecipring is to incorporate fish-based ingredients, particularly catfish, to increase its protein content (Murillo et al., 2023). Catfish has a relatively high fat content, which can increase the fat levels in cassava kecipring and may affect its stability during storage. In addition to catfish, other ingredients like gotu kola leaves can be added to enhance the product's qualities (Phoemsapthawee et al., 2022). Pegagan leaves are known for their powerful antioxidant properties, which are expected to help preserve the stability of the fortified kecipring.

Catfish is a source of essential fatty acids, including lysine and leucine, and is rich in omega-3 and omega-6 fatty acids (Trushenski et al., 2020). These nutrients significantly affect the stability of cassava kecipring during storage. The production process for cassava kecipring involves deep-frying, which uses cooking oil as a heat transfer medium. This method increases the likelihood of degradation of free fatty acids, especially in formulations that contain catfish and pegagan leaves, both of which are sensitive to high temperatures (Lis Rostini, 2016). Among the various indicators of quality deterioration, aroma is the most noticeable. Unpleasant changes in aroma can be easily perceived by consumers and directly impact the product's acceptability.

During storage, cassava crackers kecipring experience a reduction in fat content due to the production of free fatty acids resulting from hydrolysis. High levels of free fatty acids in food products signal a decrease in quality and are closely linked to the onset of oxidation for product (Ayu et al., 2016). According to the Indonesian National Standard (SNI 01-4305-1996), the maximum allowable level of free fatty acids in cassava chips is 0.7% (w/w). Oxidation, a primary form of fat deterioration, occurs from hydrolysis and oxidation. The extent of rancidity can be evaluated using the thiobarbituric acid (TBA) value and free fatty acid (FFA) analysis. FFA analysis is commonly employed to quantify the degree of lipid hydrolysis and to assess the amount of product deterioration due to this process (Fortuna Ayu et al., 2017). An increase in FFA levels clearly indicates lipid degradation and rancidity in food products. Therefore, evaluating kinetic of FFA in cassava kecipring fortified with catfish and gotu kola leaves is essential to determine quality stability throughout the storage period.

Kinetic evaluation of free fatty acid (FFA) formation was carried out in cassava cracker (kecipring) fortified with catfish and *Centella asiatica* (gotu kola) leaves. The analysis was performed using the Arrhenius model by storing the product at elevated temperatures to accelerate quality degradation. Shelf life estimation was then determined based on kinetic parameters derived from FFA accumulation as the main indicator of lipid hydrolysis. Although cassava crackers are a popular traditional snack in Indonesia, they are generally low in protein and susceptible to quality deterioration during storage. Fortification with catfish and *Centella asiatica* leaves enhances nutritional value but simultaneously increases lipid content, making the product more prone to oxidative rancidity. Limited studies have focused on the kinetic modeling of lipid degradation in fortified cassava-based products, particularly using Arrhenius approaches. Therefore, this study aimed to evaluate the kinetics of FFA formation in fortified cassava cracker during storage and to estimate its shelf life. The novelty of this research lies in applying reaction kinetics to characterize rancidity in a traditional fortified snack, providing valuable insights into its oxidative stability and storage behavior.

## 2. Methods

### 2.1. Material

The main ingredients included fresh cassava (*Manihot esculenta*), catfish (*Clarias* sp.), *Centella asiatica* leaves (known as pegagan), garlic (*Allium sativum*), shallots (*Allium cepa* var. *aggregatum*), salt, and sugar. These ingredients were sourced from traditional Bogor, Indonesia markets to reflect local food sources. The packaging material used was polypropylene plastic with a thickness of 0.4 mm. Analytical-grade chemicals, such as distilled water, 96% ethanol, phenolphthalein indicator, and a 0.1 N sodium hydroxide solution, were purchased from a chemical supply store in Bogor, Indonesia.

Sample preparation was conducted using standard kitchen equipment, including knives, gas stoves, frying pans, plastic containers, blenders, and graters. The laboratory analyses were performed with standard instruments, such as an analytical balance ( $\pm 0.0001$  g, Ohaus, USA), an incubator (Memmert IN30, Germany), an oven (Binder FD 56, Germany), a spectrophotometer (UV-1800, Shimadzu, Japan), a thermometer (IKA, Germany), and a desiccator (Kartell, Italy). Glassware, including Erlenmeyer flasks, volumetric flasks, burettes, beakers, and droppers, was supplied by Pyrex (USA). All instruments were calibrated before use.

### 2.2. Processing of Cassava Kecimpring

The cassava kecimpring, fortified with catfish and gotu kola leaves, was fried at 170°C for 30 seconds. After frying, the product was dried using a tray dryer at 72°C for 5 hours. The production process of the cassava kecimpring consisted of three main stages: processing catfish meat, preparing pegagan leaves, and formulating the fortified kecimpring. In the first stage, catfish meat was processed by separating the flesh from the bones, head, internal organs, and skin. The fish fillet was measured to be 10% of the weight of the cassava and then blended to create a smooth paste. The second stage involved preparing the pegagan leaves. The leaves were weighed to be 5% of the weight of the cassava, washed thoroughly, and then blanched at 100°C for 90 seconds. After blanching, the leaves were dried and cut into approximately 3 x 3 mm pieces the method modified (Lis Rostini, 2016).

### 2.3. Chemical Characteristics of Cassava Kecimpring

Moisture content, ash content, protein content, and free fatty acid were determined according to the method describe by (AOAC, 2005). Total carbohydrates were calculated by difference.

### 2.4. Storage Conditions for Cassava Kecimpring

Kecimpring products, packaged in 0.4 mm-thick polypropylene plastic, were stored at room temperature (25 °C) and in incubators at 35 °C and 45 °C for 28 days (4 weeks). On day 0, the fortified kecimpring products were tested to establish their initial characteristics. Subsequent tests were conducted every 7 days, specifically on days 0, 7, 14, 21, and 28 to measure free fatty acids quality modified method (Jaimez-Ordaz et al., 2019).

### 2.5. Kinetic of Free Fatty Acid of Cassava Kecimpring

Determination of the reaction order was conducted by comparing the coefficient of determination ( $R^2$ ) values obtained from the linear regression equations at each storage temperature. For zero-order reactions, the data for each quality parameter (free fatty acid content and aroma hedonic scores) were plotted with storage time on the x-axis and the quality parameter value on the y-axis. The natural logarithm of the quality parameter values ( $\ln k$ ) for first-order reactions was plotted against storage time. The reaction order corresponding to the higher  $R^2$  value was selected for each parameter (Jaimez-Ordaz et al., 2019). The slope (b) of the regression equation represents the reaction rate constant (k) of quality degradation.

Regression equation:

$$y = bx + a$$

Where:  $y$  = product quality value;  $a$  = initial quality value at the beginning of storage;  $x$  = storage duration (days);  $b$  = rate of quality change. The  $k$  value (slope) of the regression equation of each parameter with the selected order is then entered into the Arrhenius equation by plotting  $\ln k$  on the y-axis and  $1/T$  in Kelvin units on the x-axis.

$$\ln k = \ln k_0 - \left(\frac{E_a}{R}\right) \times \left(\frac{1}{T}\right)$$

Where:  $k_0$  = intercept;  $E_a/R$  = Slope;  $E_a$  = Activation Energy;  $R$  = The ideal gas constant is 1.986 cal/mol.K

A regression equation is formulated with a constant value of  $k_0$  and an activation energy value ( $E_a$ ). A reaction rate equation model for changes in product characteristics ( $K$ ) is also created.

$$K = k_0 \times e^{\left(-\frac{E}{RT}\right)}$$

The parameter with the lowest activation energy calculates the shelf life. The shelf life of kecipring is determined using the reaction kinetic equation according to the order of the response employed.

## 2.6. Statistical Analysis

The data from the free fatty acid levels were analyzed using analysis of variance (ANOVA). The interpretation of results relied on a p-value of less than 0.05, which indicates a significant effect of the treatment. Follow-up testing was performed using Duncan's test at a 95% confidence interval ( $\alpha = 0.05$ ). Microsoft Excel was used to plot quality parameter values against storage time to determine shelf life to determine shelf life. This graph included linear regression and  $R^2$  values for each storage temperature. The product's shelf life was established based on the reaction order of the chosen test parameters.

## 3. Result and Discussion

### 3.1. Result

#### 3.1.1. Characteristics of Cassava Kecimpring

Tabel 1 Characteristics of cassava kecipring

Quality Attribute (%)	Value	Standard of SNI 8646 : 2018
Moisture content	3.79	Max. 4
Ash content	0.06	Max. 0,3
Protein content	5.11	Min. 2
Fat content	19.84	Max. 30
Carbohydrate content	71.27	-

3.1.2. Free Fatty Acid of Cassava Kecimpring

Table 2 Free fatty acid of cassava kecimpring

Storage Period (Days)	Temperature storage (°C)			Average
	25	35	45	
0	0.220 <sup>a</sup>	0.220 <sup>a</sup>	0.220 <sup>a</sup>	0.220 <sup>w</sup>
7	0.399 <sup>b</sup>	1.873 <sup>b</sup>	2.272 <sup>b</sup>	1.515 <sup>x</sup>
14	0.480 <sup>b</sup>	1.762 <sup>b</sup>	2.920 <sup>bc</sup>	1.721 <sup>x,y</sup>
21	0.857 <sup>c</sup>	2.275 <sup>bc</sup>	2.952 <sup>c</sup>	2.028 <sup>y</sup>
28	1.903 <sup>d</sup>	3.065 <sup>c</sup>	3.387 <sup>c</sup>	2.785 <sup>z</sup>
Average	0.772 <sup>p</sup>	1.839 <sup>q</sup>	2.350 <sup>r</sup>	

Different superscript letters (w, x, and z) indicate significant difference by factor of storage period (p < 0.05)

Different superscript letters (p, q, and r) indicate significant difference by factor of temperature storage (p < 0.05)

3.1. 3. Kinetic of Free Fatty Acid of Cassava Kecimpring Table 3 Determination of reaction order

Temperature (°C)	Regression Equations		Determination Value	
	0 order	1 order	0 order	1 order
25	y = 0.0186x + 0.2119	y = 0.0726x – 1.195	0.2335	0.9632
35	y = 0.0596x + 0.4965	y = 0.078x – 0.4115	0.8927	0.6765
45	y = 0.0727x + 0.8238	y = 0.0818x – 0.253	0.8785	0.6150

Table 4 Estimation activation energy (EA)

Critical parameters	Regression Equations	Ea/R	Activation energy (KJ/mol)
Free fatty acid	y = -6506.3x + 17.997	6506.3	54.0119

3.2. Discussion

3.2.1. Characteristics of Cassava Kecimpring

In this study, we initially characterized the product quality to evaluate how well kecimpring complies with the standards set forth in SNI 8646:2018 for ready-to-eat fish, shrimp, and mollusk crackers. The results of this quality characterization are displayed in Table 1. The analysis results presented in Table 2 indicate that the product contains 3.79% moisture, 0.06% ash, 5.11% protein, and 19.84% fat. These values comply with the quality standards specified in SNI 8646:2018 for ready-to-eat fish, shrimp, and mollusk crackers. A notable protein and fat content increase was observed in the cassava kecimpring enriched with catfish and gotu kola leaves. This increase is primarily attributed to the high protein content of the catfish, which contributed to the elevated protein levels in the enriched product (Canti et al., 2025). Additionally, the higher fat content results from the catfish's natural lipid composition, leading to an overall increase in the fat content of the cassava kecimpring (Murillo et al., 2022).

### 3.2.2. Free Fatty Acid of Cassava Kecimpring

Products that contain fat are prone to spoilage due to oxidation (Cui et al., 2020). In cassava kecingpring, rancidity is primarily caused by the auto-oxidation of fats, which happens when the product is exposed to oxygen during processing or has residual oxygen trapped in its packaging (Laftani et al., 2019). Additionally, fat hydrolysis occurs when the product's water content leads to fat breakdown into free fatty acids. This process forms unpleasant-smelling compounds such as hydrocarbons, ketones, alkanals, and alkanols. To assess the extent of food spoilage due to rancidity, a Free Fatty Acid (FFA) analysis can be performed (Osawa et al., 2007).

An increase in free fatty acid (FFA) content was observed throughout the storage period, with the most significant rise occurring at 45 °C, followed by 35 °C and 25 °C. This trend is attributed to accelerated hydrolytic rancidity at higher storage temperatures. Specifically, 40 °C has been identified as the optimal temperature for lipase activity (Murillo et al., 2022), intensifying hydrolysis. These findings align with (Jaimez-Ordaz et al., 2019), who reported that prolonged storage significantly increases FFA levels in fortified kecingpring. During storage, residual moisture in the product promotes lipid hydrolysis, forming free fatty acids and glycerol. The accumulation of free fatty acids contributes to developing off-odors, commonly called rancidity.

### 3.2.3. Kinetic of Free Fatty Acid of Cassava Kecimpring

One factor affecting food quality is the temperature at which it is stored. Chemical reactions occur more quickly at higher temperatures (Capriles et al., 2009). The results of free fatty acid content were essential parameters for calculating the kinetics of the product. The reaction order was determined for each storage temperature by regressing the  $k$  value for the zero-order reaction and the natural logarithm of the  $k$  value for the first-order reaction (Zang et al., 2023). The reaction order was established based on the highest coefficient of determination ( $R^2$ ) obtained from the regression results.

In a zero-order reaction, the rate of food spoilage does not depend on the concentration of the reactant. Conversely, in a first-order reaction, the spoilage rate is directly proportional to the concentration of the reactant. According to (Maity et al., 2012), processes involved in food spoilage that follow zero-order kinetics include enzymatic degradation, non-enzymatic browning reactions, and fat oxidation reactions. Examples of these processes can be seen in the increased rancidity of snacks, dry foods, and frozen foods. The activation energy ( $E_a$ ) is the minimum energy required for a chemical reaction. The critical parameter with the lowest  $E_a$  value calculates a product's shelf life. A low activation energy value signifies a faster reaction rate, which leads to quicker product deterioration (Jaimez-Ordaz et al., 2019). In summary, the lower the activation energy, the more rapid the reaction will occur, resulting in faster product deterioration.

A positive activation energy value indicates that the rancidity process requires a certain amount of energy for the reaction to proceed. This phenomenon is linked to the reaction's transition state, which exists at a higher energy level than the reactants, resulting in a positive activation energy. According to (Zokti et al., 2016), food products can be classified into three categories based on their activation energy: high activation energy (50–100 kcal/mol), moderate activation energy (15–30 kcal/mol), and low activation energy (2–15 kcal/mol). Based on this classification, catfish and pegagan-fortified kecingpring fall into food products highly susceptible to rancidity. This vulnerability arises because the rancidity reaction can occur without extreme environmental conditions. Rancidity may develop at room temperature due to the breakdown of fatty acid bonds in the presence of oxygen, moisture that facilitates lipid hydrolysis, and catalytic agents like lipase enzymes, which accelerate hydrolytic rancidity reactions (Fortuna Ayu et al., 2017).

#### 4. Conclusion

The study showed that fortifying cassava crackers (kecimpring) with catfish and *Centella asiatica* leaves improved their nutritional value and made them more prone to lipid oxidation during storage. Monitoring free fatty acids (FFA), which indicates rancidity, revealed that FFA accumulation followed zero-order kinetics. The Arrhenius model determined the activation energy ( $E_a$ ) to be 54.01 kJ/mol. Higher storage temperatures significantly accelerated oxidative deterioration, demonstrating that temperature was a critical factor affecting the stability of the product. These findings emphasized the importance of proper storage conditions and the potential incorporation of antioxidants to enhance the shelf life and quality of fortified kecimpring.

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