



Kinetic Analysis of Saponification Reaction in Eco-Friendly Soap Production Based on Waste Cooking Oil

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Abstract

This study aims to determine the kinetics of the saponification reaction in making solid soap from wasted cooking oil and its effect on the quality of the solid soap produced. The novelty of this study lies in evaluates the effect of different NaOH concentrations and reaction durations on the the kinetic model with quality parameters (free fatty acid, free alkali content, and pH) to demonstrating that a pseudo second order reaction mechanism contributes to superior soap characteristics. The method begins with the refining of wasted cooking oil using activated carbon, making solid soap from refined oil with variations in NaOH concentrations of 1; 1.5; and 2 M in 30; 45; and 60 minutes. Determination of reaction kinetics was carried out by titrimetry and testing the quality of solid soap with parameters of free fatty acid content, free alkali, and degree of acidity (pH) based on SNI 3532-2021. The results showed that the saponification reaction followed 2nd order pseudo kinetics. This is supported by the value of the reaction constant reaching 1.0995 min^{-1} and the coefficient of determination $R^2 = 0,9954$. The optimum variation of solid soap obtained is 2 M NaOH concentration in 60 minutes. Soap products produced through pseudo second-order reaction kinetics have high quality with a white appearance, no rancid odor, smooth and soft texture. This is evidenced by the parameters of free fatty acid and free alkali content of 0.1126% and 0.016%. The degree of acidity (pH) of the solid soap produced is also in the range of 9.5-10. All of these parameters have met the criteria for solid soap according to SNI 3532: 2021.

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INTRODUCTION

Amid growing environmental awareness, waste cooking oil generated from household and food industry activities has become an increasingly pressing global issue. According to data from the Ministry of Environment and Forestry (2022), over 1,6 million tons of waste cooking oil are disposed of annually without proper treatment, leading to severe environmental impacts and significant harm to public health. An alternative approach has emerged to address this problem by converting waste cooking oil into eco-friendly soap through the saponification process. This process involves a chemical reaction between triglycerides in the oil and an alkali such as sodium hydroxide (NaOH) (Xiaojie *et al.*, 2022). This method not only reduces waste but also produces biodegradable soap that is safer for the environment compared to conventional soap.

Understanding the kinetics of the saponification reaction is crucial for optimizing the production process of eco-friendly soap. Factors such as temperature, alkali concentration, oil to alkali molar ratio, and stirring time affect the reaction rate. Research by Mardawati *et al.*

(2019) has shown that the free fatty acid content in waste cooking oil can increase the reaction rate but may also reduce the quality of the resulting soap. While a faster reaction is possible, excessive free fatty acid levels result in soap that does not meet desired quality standards.

In various studies, the saponification reaction is often analyzed using a second order kinetic model, where both reactants significantly influence the reaction rate. However in practice, the alkali concentration is typically much higher than that of the fatty acid reactants. Research by Çıtak and Kırak (2015) has reported that the second-order rate constant is very low, only $0.0038 \text{ L mol}^{-1} \text{ s}^{-1}$, indicating a very slow reaction. Similarly, research by Low (2017) found that minimal increases in the reaction rate constant despite significant temperature increases, underscoring the inadequacy of this model for industrial applications. Higher alkali concentrations are thus required to optimize soap formation rates.

Despite extensive studies on saponification kinetics, there remains a lack of understanding regarding how the specific characteristics of waste cooking oil affect the reaction rate. Most existing research has concentrated on pure vegetable oils, with limited application of kinetic models to waste-derived oils. This gap hinders the optimization of saponification processes using more sustainable, recycled feedstocks (Gultom et al., 2024). Therefore, this study addresses the need to evaluate the kinetics of saponification using waste cooking oil, with a focus on identifying the most suitable reaction order models to enhance soap quality and process efficiency.

METHOD

Instruments and Materials

Instruments

The equipment used in this study includes a balance, beaker, bulb, burette, clamp, dropper pipette, Erlenmeyer flask, filter paper, glass funnel, graduated cylinder, hot plate, magnetic stirrer, Ostwald viscometer, pH meter, pycnometer, spatula, stand, stirrer, stirring rod, and volumetric flask.

Materials

The materials used in this experiment include activated carbon, distilled water (H_2O), ethanol ($\text{C}_2\text{H}_5\text{OH}$), phenolphthalein indicator, sodium hydroxide (NaOH), starch solution, and wasted cooking oil.

Purification of Wasted Cooking Oil

The purification of wasted cooking oil was carried out based on a modified method from the study by Kurniawan et al. (2020). Initially, 250 grams of waste cooking oil were weighed and placed into a 500 mL beaker. The oil was then heated to 70°C . To the heated oil, 10 grams of activated carbon were added and followed by the introduction of a starch solution. The mixture was stirred continuously using a mechanical stirrer for 60 minutes, then heated to 150°C to facilitate the formation of a suspension. After allowing the mixture to rest for 24 hours, it was filtered using filter paper to remove impurities and improve the oil's color. The resulting purified oil was then ready for use in soap production.

Production of Solid Soap

The process of solid soap production was determined by modifying procedure of Khuzaimah (2018). It begins with preparing 25 mL, 37 mL, and 50 mL portions of the purified oil in separate beakers. Sodium Hydroxide (NaOH) solution is prepared by weighing 4, 6, and 8 grams of NaOH and dissolving each in 100 mL of distilled water, yielding concentrations of 1,

1.5, and 2 M. The purified oil is heated to a processing temperature of 50°C. Each variation of NaOH solution is then added gradually to the heated oil while stirring and continuing to heat the mixture on a hot plate until it reaches 70°C. The mixture is allowed to react for 30, 45, and 60 minutes until it thickens.

Reaction Kinetics Determination by Titrimetry

This step was carried out based on an adapted procedure from Pasaribu (2022). The mixture of purified oil and NaOH from the previous step is stirred until the two solutions react completely. Once the reactants are mixed, 10 mL of the solution is taken and added to an Erlenmeyer flask containing 10 mL of standard 0.1 N HCl solution. The mixture is stirred, and 3 drops of phenolphthalein indicator are added. The solution is then titrated with 0.1 N NaOH until the endpoint is reached, indicated by a pink color. The volume of titrant used is recorded. This procedure is repeated for reaction times of 30, 45, and 60 minutes.

Free Fatty Acid and Free Alkali Content in Soap

The determination of free fatty acid and free alkali content in soap was conducted according to SNI 3532-2021 (SNI, 2021). A sample of 5 grams soap was weighed for each variation and placed into an Erlenmeyer flask, followed by the addition of 50 mL of warm ethanol. Three drops of phenolphthalein indicator were added to the solution, which was then titrated with 0.1 N HCl until the pink color disappeared. The volume of HCl used for each variation was recorded, the free fatty acid and free alkali content in the soap was determined. The equations used to determine the percentage of free fatty acids (%FFA) and free base alkali (%FBA) are based on the guidelines stated in SNI 3532:2021 (SNI, 2021).

$$\% \text{FFA} = \frac{V_{\text{HCl}} \times N_{\text{HCl}} \times \text{MW Oleic Acid}}{W \times 1000} \times 100\%$$

$$\% \text{FBA} = \frac{V_{\text{HCl}} \times N_{\text{HCl}} \times \text{MW NaOH}}{W \times 1000} \times 100\%$$

Description:

V NaOH = Volume of NaOH Solution (mL)

N NaOH = Normality of NaOH (N)

MW Oleic Acid = 282 g/mol

MW NaOH = 40 g/mol

W = Sample Weight (g)

Acidity Level (pH)

The pH test was conducted by SNI 3532-2021 (SNI, 2021). The procedure began by weighing 1 gram of soap, then adding 100 mL of distilled water into a beaker and stirring until homogeneous. The pH of the solution was measured using a pH meter that had been calibrated with pH 7 and 10 buffer solutions. The pH meter was immersed into the beaker containing the soap solution and allowed to stabilize until the pH value remained constant. This procedure was repeated for each variation and the obtained pH values were recorded.

RESULTS AND DISCUSSION

Saponification Reaction Kinetics in Solid Soap Production

The saponification reaction is a chemical process in which fatty acids or oils (triglycerides) react with a strong base such as sodium hydroxide (NaOH) to produce soap and glycerol. Triglycerides consist of one glycerol molecule bound to three fatty acid molecules through

ester linkages. When a strong base like NaOH is added to the triglyceride mixture, an ester hydrolysis reaction occurs. NaOH acts as a breaking agent for the ester bonds connecting glycerol and fatty acids. This causes the triglyceride to break down into fatty acids that bind with sodium ions (Na^+), forming a fatty acid salt known as soap, while glycerol is produced as by product (Puspitasari *et al.*, 2023).

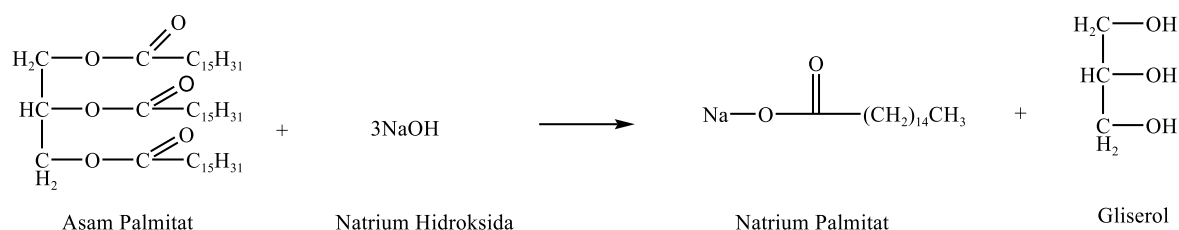


Figure 1. Saponification Reaction in Soap Production

During the hydrolysis process, chemical energy is released which results an increase in the temperature of the reaction mixture. This temperature increase accelerates the reaction rate because the particles in the solution move more quickly, created an entropic condition that increases the frequency of molecular collisions and speeds up the breakdown of ester bonds in triglycerides. The heat generated can increase the solubility of free fatty acids, making the reactants more likely to interact with each other (Khan *et. al.* 2021).

The kinetics of the saponification reaction were determined using a titration-based approach, which provided a precise means of quantifying the residual sodium hydroxide (NaOH) remaining in the reaction mixture. This method adheres to the fundamental principles of reaction kinetics to track the progress of soap formation. Hydrochloric acid (HCl) was introduced as a neutralizing agent to react with unconsumed hydroxide ions (OH^-), completing the reaction and producing water (H_2O) as by product. By neutralizing the excess NaOH, the volume of HCl added became a direct measure of the unreacted NaOH present in the system. A subsequent titration with NaOH was conducted to determine its remaining concentration in the reaction medium. Changes in NaOH concentration offered valuable insights into the reaction's progression: a decreasing trend indicated significant advancement of the reaction, while a plateau or increase suggested a slowing of the reaction rate over time (Pasaribu, 2022).

Table 1. Values of $1/[\text{A}]$ and $1/[\text{B}]$ Against Time Variations Using the Titration Method

t (minutes)	V_{NaOH} (mL)	$[\text{NaOH}]$ (M)	$[\text{Oils}]$ (M)	$1/[\text{A}]$	$1/[\text{B}]$
0	25	0,04	0,0042	25	238,0952
30	28	0,0354	0,0036	28,2486	273,2240
45	30	0,0335	0,0034	29,8507	290,6977
60	31	0,0327	0,0033	30,5810	303,0303

The research results indicate that the longer the saponification reaction is allowed to proceed, the greater the volume of NaOH titrant required, as detailed in Table 1. In the initial stages of the reaction, hydroxide ions (OH^-) from NaOH react with triglycerides to produce soap and glycerol, causing a significant decrease in NaOH concentration. As the reaction progresses, most of the triglycerides have already undergone reaction, leaving less NaOH available to be consumed. This trend demonstrates that prolonged reaction times lead to a reduction in the availability of OH^- ions, as the majority have been utilized in the formation of soap and glycerol. Consequently, the concentration of free OH^- ions in the solution diminishes over time. As a result, a larger volume of NaOH is required during titration, since the remaining unreacted base must be neutralized by (Pasaribu, 2022).

Based on the calculations, the values of $1/[\text{A}]$ for second-order reaction kinetics and $1/[\text{B}]$ for pseudo-second-order reaction kinetics were obtained for each time variation of 0, 30, 45, and

60 minutes as illustrated in Figure 2. These kinetic models are commonly used to describe the saponification process when considering heterogeneous systems involving oils and alkaline solutions (Wangi *et al.*, 2024). Second-order kinetics typically apply when both reactants influence the rate, whereas pseudo-second-order models are often employed to simplify complex reaction mechanisms when one reactant is in large excess (Laborda *et al.*, 2024).

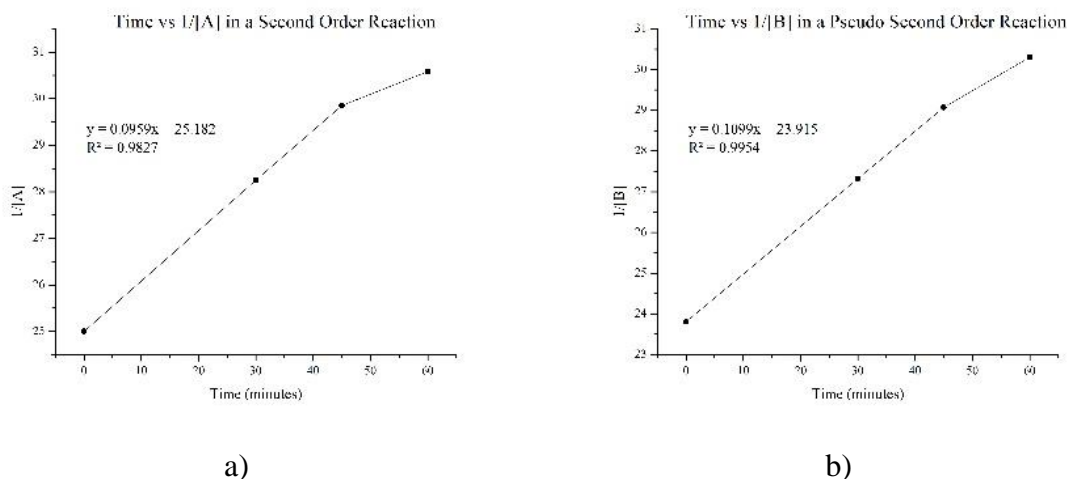


Figure 2. a) Graph of Time Variation vs. $1/[A]$ for Second-Order Reaction; b) Graph of Time Variation vs. $1/[B]$ for Pseudo-Second-Order Reaction

Graph (a) depicts the relationship between reaction time and the concentration of the NaOH reactant ($1/[A]$) in a chemical reaction hypothesized to follow second-order kinetics. The data exhibit a linear trend, with an increase over time described by the linear regression equation $y = 0.0959x + 25.182$ and a coefficient of determination $R^2 = 0.9827$. The gradient value of 0.0959 min^{-1} is interpreted as the reaction rate constant, derived from the second-order rate law equation.

$$\frac{1}{[A]} = kt + \frac{1}{[A]_0}$$

In comparison to graph (a), which analyzes the saponification reaction kinetics as a second-order process, graph (b) reveals that the reaction rate in the pseudo-second-order scenario is faster. Graph (b) shows the relationship between reaction time and the concentration of the oil reactant ($1/[B]$) in a pseudo-second-order reaction. The data shows a positive linear correlation with the regression equation $y = 1.0995x + 23.915$ and a coefficient of determination (R^2) = 0.9954. The R^2 value close to 1 indicates a high degree of accuracy in modeling the pseudo-second-order kinetics, even higher than that observed for the second-order reaction. The gradient value of 1.0995 min^{-1} suggests a higher reaction rate constant in this case compared to that of the second-order reaction in graph (a).

This difference can be explained by the much higher concentration of NaOH compared to the fatty acids (waste cooking oil) in the saponification reaction. Under pseudo second order conditions, the excess NaOH concentration remains effectively constant throughout the reaction.

$$-\frac{d[B]}{dt} = k[A][B] \Rightarrow -\frac{d[B]}{dt} = k'[B], \text{ dengan } k' = k[A]_0$$

In this equation, k' denotes the apparent rate constant, which is significantly elevated due to the substantial initial concentration of the base $[A]_0$. This factor accounts for the markedly higher rate constant observed in pseudo-second-order reactions compared to true second-order

reactions. The surplus of NaOH effectively mitigates any constraints on the reaction rate that might arise from a diminishing base concentration as the reaction progresses. Consequently, the frequency of collisions between triglyceride molecules and OH^- ions remains consistently high, thereby expediting the saponification process (Agustin and Hendrawati, 2022).

In a second-order reaction, the concentrations of NaOH and fatty acids influence the reaction rate directly. Over time, the concentration of NaOH decreases sharply, limiting the availability of OH^- ions to react with fatty acids. This reduction in concentration creates a mass transfer limitation as the frequency of collisions between NaOH particles and fatty acids diminishes, thus slowing down the reaction rate. However, under pseudo-second-order conditions, the excess NaOH alleviates this limitation, maintaining the reaction kinetics at a stable rate. In the presence of excess NaOH, fatty acids undergo complete reaction to yielding saponification products soap and glycerol. This condition not only accelerates the soap-making process but also enhances the quality of the environmentally friendly soap product (Angelia *et. al.* 2022).

Free Fatty Acid and Free Alkali Content

Free fatty acids are components of fatty acids present in soap samples but not bound as sodium compounds or triglycerides. The presence of free fatty acids in soap results from fatty acids that have not undergone saponification with bases such as NaOH. The analysis of free fatty acid content aims to determine the amount of free fatty acids present in soap products. According to SNI 3532:2021, the maximum permissible content of free fatty acids in soap should be $<2.5\%$. If the free fatty acid content exceeds this threshold, the soap tends to develop a rancid odor, a softer texture, and a darker color (Midiyarti *et. al.* 2016).

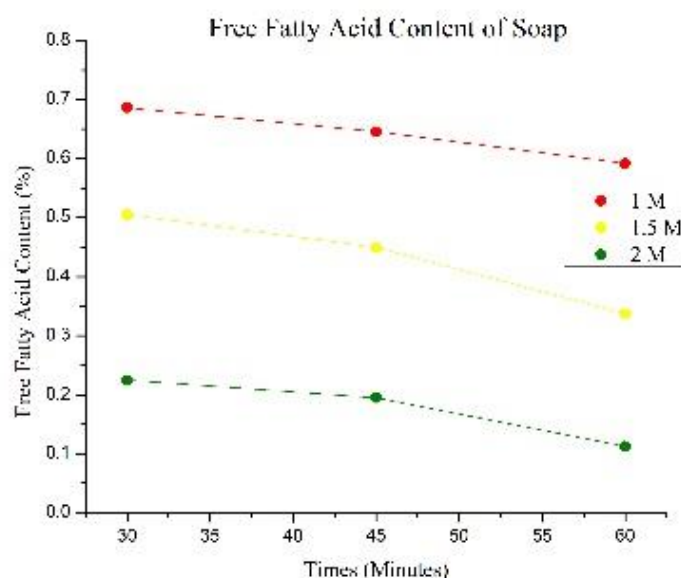


Figure 3. Graph of Free Fatty Acid Content in Soap at Different Time Variations

Based on the graph above, it is evident that the free fatty acid (FFA) content decreases as the saponification reaction time increases and the base concentration rises. At a NaOH concentration of 2 M with a reaction time of 60 minutes, the FFA content reaches the most optimal result only 0.1126%. Even at the lowest base concentration, a reaction time of 30 minutes in 1 M made the free fatty acid content remains very low at just 0.6861%. This is due to the increase in base concentration, which provides a greater number of hydroxide ions (OH^-) in the solution, thereby enhancing the collision frequency between OH^- ions and free fatty acid molecules (Salanti *et al.*, 2022).

This saponification reaction follows pseudo-second-order kinetics, where the reaction rate depends on the concentrations of fatty acid reactants and OH^- ions with the excess NaOH concentration assumed to be constant. The OH^- ion acts as a nucleophile, attacking the carbonyl group of free fatty acids, breaking the ester bond to form soap and glycerol. As the OH^- concentration increases, the rate of nucleophilic attack accelerates, thereby enhancing the formation of fatty acid salts (soap) through the saponification reaction. This results in a significant decrease in free fatty acid content over time, due to the accelerated reaction rate between OH^- ions and free fatty acids (Vicentini *et al.*, 2021)

Free alkali refers to the amount of alkali that is not bound as compounds in the soap. The presence of excess free alkali in soap may be caused by an excess base concentration during the saponification process that does not react with fatty acids. The determination of free alkali content in solid soap is intended to assess the quality of the unbound alkali in the soap, as specified by Indonesian National Standards (SNI). According to SNI 3532:2021, the maximum allowable free alkali content in soap should be $<0.1\%$. If the free alkali content exceeds this limit, the soap may cause irritation, as the strong base can damage the skin's lipid layer, making it more susceptible to inflammation (Nitbani *et al.*, 2020).

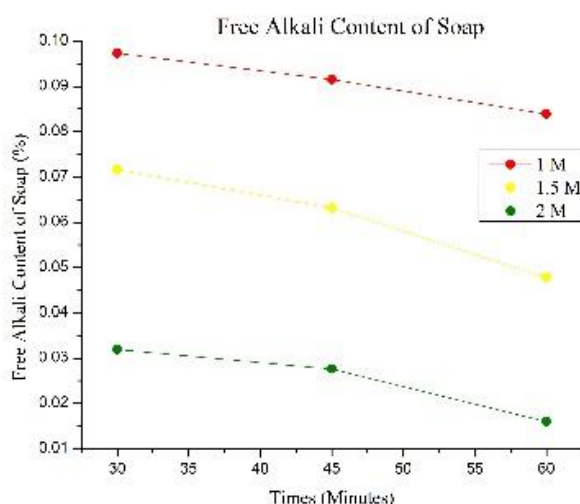


Figure 4. Graph of Free Alkali Content in Soap at Different Time Variations

The graph above illustrates the decrease in free alkali content in soap over time for each variation in concentration. The most optimal free alkali content is observed at a NaOH concentration of 2 M with a reaction time of 60 minutes, which is 0.016%. However, the NaOH concentration of 1 M with a reaction time of 30 minutes shows a relatively high free alkali content of 0.0973%, yet still falls within the parameters of SNI 3532:2021 ($<0.1\%$), thus classifying it as a high-quality soap product. This is due to the fact that at lower NaOH concentrations, the saponification reaction is not fully completed due to the limited amount of base available to react with the fatty acids, resulting in a relatively higher free alkali content (Agustin *et al.*, 2022).

The saponification reaction kinetics following pseudo-second-order behavior causes the excess base to remain unreacted after all available fatty acids have reacted, leaving residual OH^- ions in the mixture. This condition leads to a relatively high free alkali content due to the pseudo-second-order reaction kinetics. However, extending the reaction time can increase the amount of triglycerides that react, allowing for more interactions with OH^- ions to form soap. This will significantly reduce the free alkali content as the saponification reaction reaches equilibrium, resulting in a high-quality soap product (Hesni *et al.*, 2022).



Figure 5. Solid Soap Product Quality Results

Based on the image above, all variations of the produced soap exhibit high quality, with a white appearance, no rancid odor, and a smooth, soft texture. This indicates that the low levels of free fatty acids and free alkali significantly impact the quality of the solid soap produced through pseudo-second-order reaction kinetics, meeting the criteria outlined in SNI 3532:2021.

Acidity Level (pH)

The pH level is a parameter that indicates the acidity or alkalinity of the produced solid soap. The use of alkali in large quantities increases the pH value of the soap. The higher the alkali content, the higher the soap's pH. Excess alkali occurs because some of the alkali does not react with the fatty acids during the saponification process, resulting in free alkali. According to SNI 3532:2021, soap must have a pH between 8 and 11 as it is considered a weak base which optimal for emulsifying fats and dirt (Midiyarti *et al.*, 2016)

If the soap's pH is below 8, its basic properties weaken and may even become acidic, reducing the ability of OH^- ions to interact with oils and dirt. This makes the soap less effective in removing oils and dirt, as the emulsification reaction does not proceed optimally. Conversely, if the soap's pH exceeds 11, the excess OH^- ions can disrupt the skin's natural lipid barrier which is essential for moisture retention and protection against infection. The ions of OH^- in the soap can draw protons (H^+) from lipid and protein components in the skin, damaging their molecular structure. This reaction may cause protein denaturation and disturb the skin's natural pH balance (4.5–6.5) (Alum *et al.*, 2024).

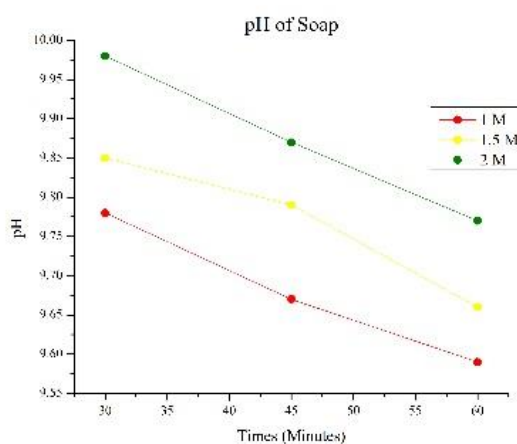


Figure 6. pH Graph of Soap at Different Time Variations

Based on the graph above, the pH values obtained range from 9.5 to 10. This indicates that the results meet the criteria outlined in SNI 3532:2021. Within this pH range, the hydroxide ions (OH^-) present in the soap work optimally to break down the fat and oil bonds on the skin's surface through molecular cleavage. The OH^- ions can decompose fats or oils via partial saponification. The OH^- ions attack the carbonyl group of fats or oils through a nucleophilic mechanism. This attack breaks the ester bond in the oils, resulting in free fatty acids and glycerol, which are water-soluble and can be easily removed from the skin (Baena *et al.*, 2022). Therefore, this process enables the soap to clean dirt and oils effectively.

CONCLUSION

Based on this study, it can be concluded that the saponification reaction for the production of solid soap from used cooking oil follows pseudo-second-order kinetics, with a reaction rate constant of 1.0995 min^{-1} and a determination coefficient (R^2) of 0.9954. Among the tested models, the pseudo-second-order model provided the best fit to the data, indicating a more accurate representation of the reaction mechanism. The resulting soap exhibited desirable characteristics soap is white in appearance, free from rancid odor, with a smooth and soft texture. The optimum result was obtained at a NaOH concentration of 2 M and a reaction time of 60 minutes, yielding a product with free fatty acid content of 0.1126%, free alkali content of 0.016%, and a pH range of 9.5–10, all of which reached the quality standards set by SNI 3532:2021. This study demonstrates that applying the pseudo-second-order kinetic model in the saponification process provides a more precise and reliable approach for predicting reaction behavior. The findings offer a useful reference for future studies and suggest that this model can be effectively used to improve the efficiency and consistency of solid soap production from waste based raw materials.

RECOMMENDATIONS

Future research should explore the scalability of this pseudo-second-order saponification reaction by optimizing reaction parameters such as stirring speed, temperature control, and the use of alternative catalysts to enhance efficiency. Additionally, investigating the impact of different waste cooking oil sources on soap quality could provide broader applicability. Further studies should also assess the long-term stability and biodegradability of the soap to ensure its environmental benefits remain consistent over time.

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