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# EFFECT OF HEATING TIME ON CHANGES IN PHYSICOCHEMICAL PROPERTIES AND FATTY ACID COMPOSITION OF RED PALM OIL

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## ABSTRACT

Crude Palm Oil (CPO) contains approximately 50% saturated fatty acids and approximately 40% unsaturated fatty acids. Unsaturated fatty acid content is known to be effective in lowering blood cholesterol levels. Palm oil also contains β-carotene which is very beneficial for health. Palm oil processing is defined by three stages, namely degumming, bleaching and deodorization. The bleaching process is carried out at 90–105°C using 1-2% bleaching earth and deodorization at 240-270°C. The high concentration of bleaching earth and deodorization temperature causes carotene to be degraded leading to low carotene content in the final product. The effort to maintain the carotene content is to process CPO into red palm oil (RPO). The content of carotene and fatty acids contained therein will be damaged in the presence of high heat, ascertain this, a study was conducted on the effect of heating time on the chemical properties of red palm oil. The study was conducted using a completely randomized block design with prolonged heating treatment (0, 2, 4, 6, 8 and 10 hours) at 180°C and repeated 3 times and then the fatty acid content, peroxide value, iodine number and functional groups formed in bulk red palm oil purchased online were observed. The results showed that increasing the heating time can decrease the iodine number but increase the acid number and peroxide value. Heating at 180°C for 10 hours produced iodine, acid and peroxide value of 57.72, 9.56 and 12.55, respectively. The results of the analysis using Fourier Transform Infra-Red (FTIR) showed the presence of free fatty acids at wave number 722 $cm^{-1}$ and peroxide numbers at wave number 2.854 $cm^{-1}$. The test results using gas chromatography showed that linoleic acid decreased by about 3.23% during heating for up to 10 hours, while saturated fatty acids increased by about 0.32%.

**Key words:** Cooking oil, Free fatty acid, Iodine number, Peroxide number, polymerization

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## INTRODUCTION

Indonesia, Malaysia, Thailand, Colombia and Nigeria are the primary producers of palm oil. Palm oil can be processed into red palm oil which is rich in carotene, which is 15 and 300 times that of carrots and tomatoes, respectively [1]. Carotenoid compounds contained in palm oil include $\alpha$-carotene, $\beta$-carotene, $\gamma$-carotene, xanthophyll and lycopene, amounting to 500-700 ppm of the saponified portion in tocopherol and lutein content [2]. Palm oil also contains vitamin E (tocotrienols), which has strong antioxidant properties and can inhibit the synthesis of cholesterol [3]. Carotenoids, tocopherols, tocotrienols, sterols, triterpenes, alcohols, phospholipids, glycolipids, terpenic hydrocarbons and aliphatic components are all present [4]. It serves as a carrier for provitamin A and vitamin E for consumers and can be used as a natural dye. It can also be produced through traditional methods and can be sold in both unbranded and branded forms. Red Palm Oil is employed as a frying medium, carried out at a temperature of 180ºC [5]. Heating for a long time can change its physicochemical characteristics [6]. However, resistance and stability to thermal oxidation vary depending on the composition of fatty acid.

Oils with high polyunsaturated fatty acids are more susceptible to thermal oxidation [7]. However, palm oil can stand high temperatures due to its high percentage of saturated bonds. Monounsaturated and polyunsaturated fatty acids are types of unsaturated fatty acids. Fatty acids are also classified as cis_ or trans_ based on the difference in molecule configuration around the double bond [8]. Heating causes changes in the composition of cooking oil due to hydrolysis, oxidation and polymerization processes to produce primary and secondary oxidation products [9]. The oil that has been damaged will cause health problems when used continuously. The palm oil fatty acid composition consists of palmitic (40.93%), stearic (4.18%), oleic (41.51%) and linoleic (11.64%) acids [10]. The acids and peroxide values are used to determine the oil quality. The acid value is regarded as a key indicator of oil oxidation. This method is measured in non-aqueous solvents using acid-base titration techniques [11]. Peroxide number (PV) is an indicator of oxidation and is important for determining the level of oil damage [12]. The iodometry method is a common method to determine PV that indicates the unsaturated fatty acids in a double bond to form peroxide. Furthermore, the peroxide number indicates oil oxidation, so the value is useful for determining quality after processing and storage. Peroxide value will increase to a certain level during storage before usage, depending on time, temperature and exposure to light and air. A high peroxides value indicates continuous oxidation, and heating initiates a chain of chemical reactions including hydrolysis, oxidation and polymerization [12]. Loganathan *et al.* [13] investigated the effect of prolonged

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heating using three common cooking techniques, namely deep-fat frying, microwave and conventional oven cooking on carotene and tocopherol profiles. Peroxide, p-anisidin, and total oxidation value, acidity and fatty acid composition were used to determine physicochemical changes in all oils. Both RPO and palm olein were found to be thermally stable due to lower levels of hydrolytic and oxidative degradation and higher tocopherol retention, allowing the oil to be heated for up to 3 hours in a deep-fat fryer and conventional oven. However, due to its lower carotene retention, red palm olein appears to be unsuitable for prolonged heating processes. The stability of phytonutrients is also affected by microwave heating [13]. Red Palm Oil which is sold in bulk on the online market is recommended as a substitute for cooking oil even though there has not been much testing regarding the possibility of oil damage if used at high temperature and frying time. The purpose of this study was to find out how much damage would occur if red palm oil was heated at 180$^{o}$C with different frying times for the fatty acid content, peroxide number, iodine number and functional groups formed in bulk red palm oil purchased in bulk online. The results of the research are expected to be able to provide recommendations for how long heating of red palm oil can still be done so that it remains safe from damage.

## MATERIALS AND METHODS

### Materials

Red Palm Oil (RPO) can be obtained through online purchases. A 1,500 mL of RPO was heated in a Pyrex beaker at 180ºC for 0, 2, 4, 6, 8 and 10 hours. The heated samples were allowed to cool at room temperature for 24 hours. Samples were taken for analysis after each heating and cooling cycle and stored in a freezer (-4 $^{o}$C) until required for further tests. Peroxide number, iodine value, and Free Fatty Acid (FFA) were then determined using a estándar method base don AOCS method Ce 2-66 [15].

### Fourier Transform Infra-Red (FTIR) spectroscopic analysis

The FTIR instrument used was Agilent's Carry 639 FTIR brand. Before drying with lens tissue, a KBr disk was cleaned and rinsed with pure n-hexane to remove any remaining residue from the oil sample. After that, it was measured at wave numbers between 4,000-400 cm$^{-1}$ with a resolution of 1.9 [16].

### Test method for saturated and unsaturated fatty acids by gas chromatography

The FAMEs were determined by GC (Clarus 580 Saturated Fat) [17]. The instrument measurement conditions showed a split injection volume and temperature of 1.0 L and 240ºC, respectively. The carrier gas used was helium with a temperature gradient and detector of 50-230°C and 240ºC. Meanwhile, the

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$H_2$ and air flows were 30 and 300 mL/min, respectively, and the FAMEs were analyzed by comparing their retention times to the required standard.

### Statistical analysis

In the six treatments analyzed, red palm oil was heated at 180°C for 0, 2, 4,6, 8 and 10 hours in 3 replications. The study was conducted using a completely randomized block design. The data were analyzed by the use of analysis of variance and additional tests with the smallest significant difference ($p < 0.05$) was utilized for testing on data of peroxide number, iodine value and acid value.

## RESULTS AND DISCUSSION

### Iodine Value

The analysis of variance confirmed a sizable difference among the heating time at ($p<0.05$). The decrease in the oil's iodine value is caused by a more intense thermo-oxidative transformation, indicating an increase in the oxidation level. It can be associated with oxidation and polymerization reactions involving double bonds [18]. Palm oil has an iodine number between 44-54, and the heating time causes a decrease as shown in Figure 1. This number can decrease due to the breaking of unsaturated bonds to become saturated due to heating or frying. Heating oil causes oxidation, hydrolysis, and decomposition of oil, influenced by the high temperature and duration of heating [19].

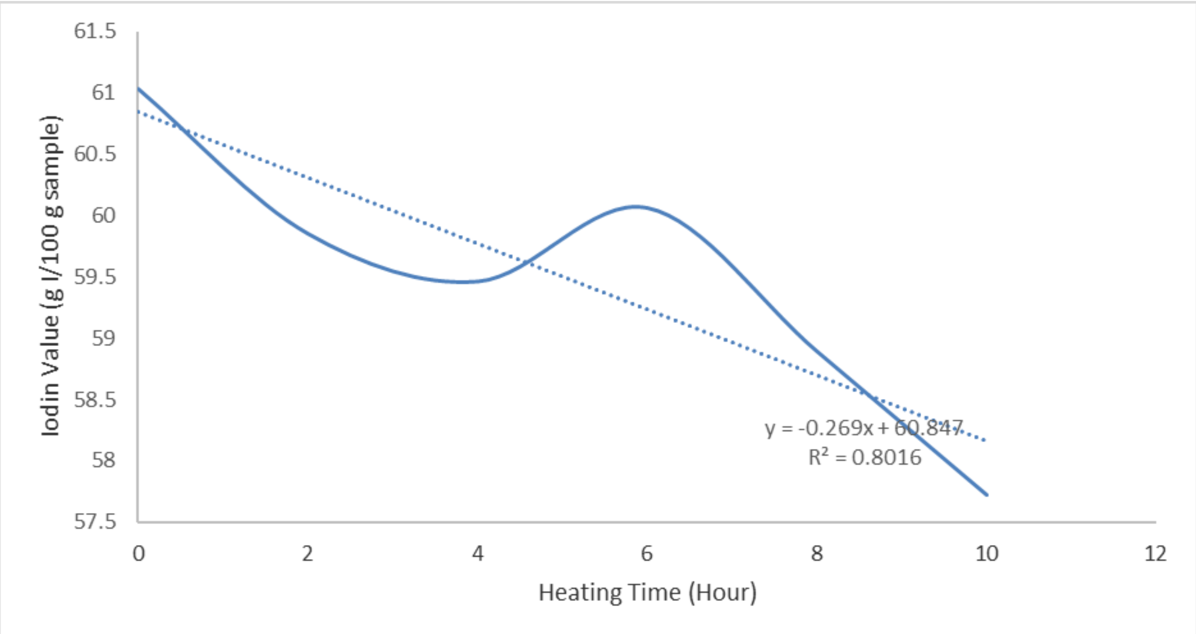


**Figure 1: Iodine Number Value due to different heating times**

The mixture of soybean oil and hydrogenated vegetable oil experiences a slight increase in the percentage of saturated fatty acids during frying [20]. Other studies have discovered an increasing level with increasing frying time [21]. As a result, oxidation and polymerization reactions can increase saturated fatty acid levels proportionally [22]. According to Indonesian National Standard, good quality cooking oil has an iodine value between 7.7 - 10.5 mg/100g [23]. Red palm oil analyzed in this study had an iodine value higher than the SNI standard, this is due to the presence of carotenoids containing double bonds.

**Acid Number**

The results showed significant differences ($p < 0.01$) in the acid number of the oil, which can be increased by the heating time (Figure 2).

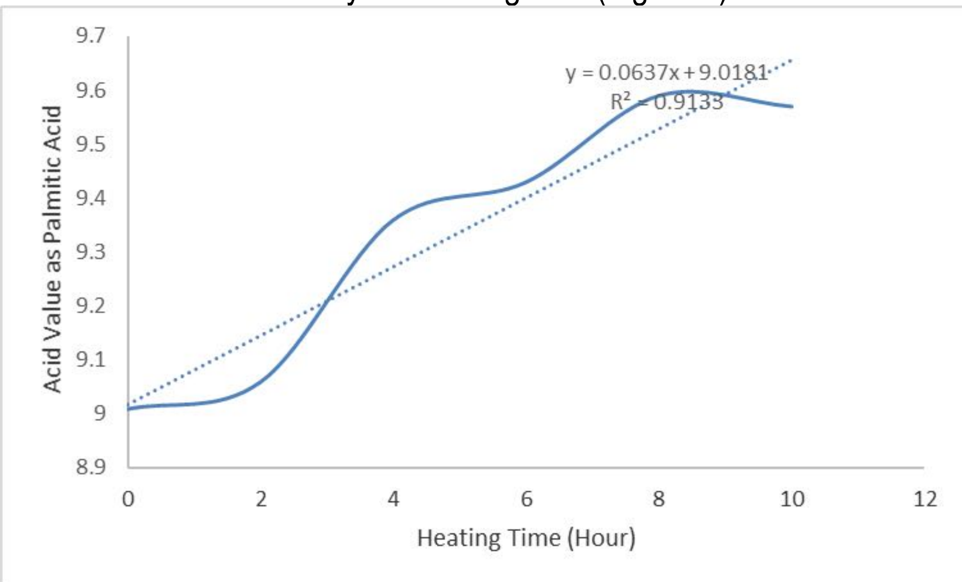


**Figure 2: Effect of heating time on the acid number of red palm oil**

Acidity is an indicator that can be used to assess the hydrolytic and oxidative stability of samples. The process of oxidation and chemical changes in oil is seen with increasing free fatty acid content and decreasing total unsaturation. The fatty acids influence oxidative stability, which is its main component. According to Jadhav *et al.* [22], the acid value of palm oil appears to increase during the frying cycle. This study's palm oil contained 40% oleic acid, 43% palmitic acid, 6% stearic acid and 10-12% linoleic acid. Oleic acid is very resistant to oxidative damage during frying, where the higher quantum of oleic acid makes the oil more resistant to oxidative degradation than polyunsaturated fatty acids, giving it a slight increase in acid value. According to the Indonesian National Standard 3741:2013, cooking oil should have a free fatty acid content not exceeding 0.6 mg NaOH/g [23]. However, all red palm oil does not meet the Indonesian SNI standard for acid value.


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**Peroxide Number**

The peroxide number increased significantly (p0.05) as heating time increased (Figure 3). Nduka *et al.* [24] demonstrated that heating time increased the peroxide value of palm oil olein.

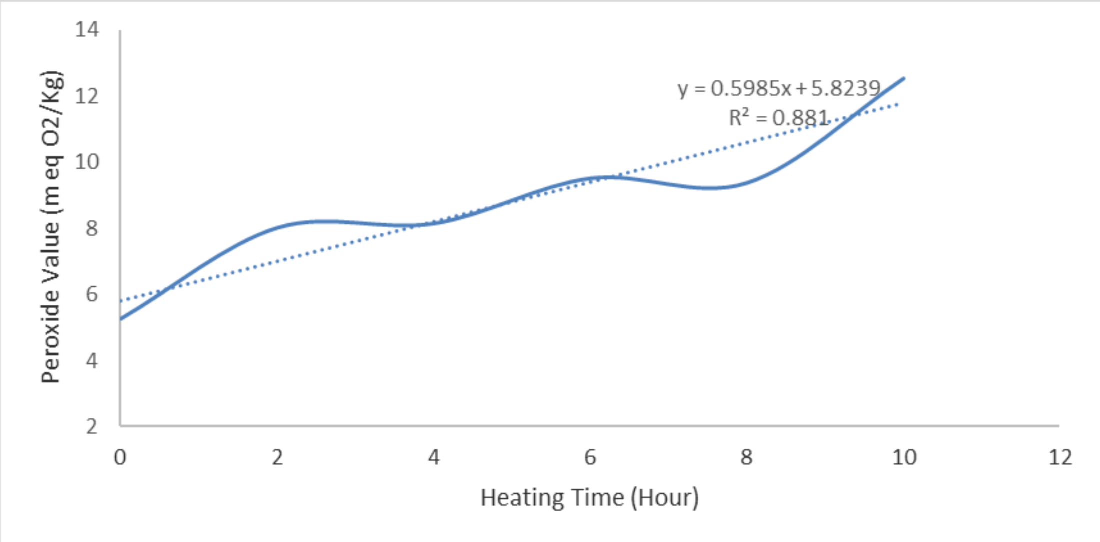


**Figure 3: Effect of heating time on the peroxide value of red palm oil**

Peroxide numbers increased at the beginning of heating [18]. In this study, the increase occurred up to the first 10 hours. This is almost the same as the results of research using a mixture of palm oil and peanut oil, while a mixture of palm oil and soybean oil obtained fluctuation data. This is due to the decomposition of peroxides produced during the primary secondary oxidation process [25]. This amount will be reduced due to the peroxide decomposition into secondary oxidizing compounds. Several studies have observed a decrease with increasing heating time. This is because the peroxides formed during heating are unstable to high temperatures and frying time, converting them to carbonyls and aldehydes [26]. Palm kernel oil > peanut oil > palm olein oil > soybean oil is the trend before and after heating. When heated oil was compared to fresh Arachis oil, Falade and Oboh [6] discovered a significant increase in acid and peroxide values, as well as malondialdehyde concentrations. Subsequently, Koh and Surh [27] found that peroxides decomposed more rapidly with increased temperature, and hydroperoxides formed at ordinary frying temperatures give secondary products. As a result, this parameter is not ideal for determining the decomposition status of palm oil. Many studies have demonstrated the effect of temperature on lipid decomposition. The effect is drastic once the temperature exceeds 200$^{o}$C since the recommended standard is 180$^{o}$C. For the first stage of the oxidation process, determining acidity and peroxide value separately is preferable. According to the Indonesian National Standard 3741:2013, good quality cooking oil has a maximum


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peroxide value of 10 meq $O_2$/kg, thus above 10 meq $O_2$/kg, indicating spoilage [28]. The overall treatment showed that red palm oil with heating for 0 to 8 hours still met the requirements of the Indonesian National Standard.

**Fourier Transform Infra-Red Assessment**

Fourier Transform Infra-Red (FTIR) spectroscopy can identify specific functional groups in samples. A certain absorption band identifies them at a particular wavenumber ($cm^{-1}$), and the amount of absorption may vary between samples. Cis and trans (-C=C-) double-bonded alkene functional groups, C = O esters, C-H methyl, and secondary oxidizing compounds are represented by the FTIR peaks and wavenumbers. Meanwhile, several functional groups from the spectrum of vegetable oils have been identified, including esters, carboxylic acids and saturated and unsaturated hydrocarbons [16, 25, 29,30]. Alkenes (-C=C-) are common functional groups in vegetable oils, and the high percentage of unsaturated fatty acids corresponding to the wavenumbers were discovered 722 $cm^{-1}$, 966 $cm^{-1}$, 1,654 $cm^{-1}$, 1,418 $cm^{-1}$, and 3,005.54 $cm^{-1}$ [30]. Moreno *et al.* [31] used a wave number of about 3,006 $cm^{-1}$ to determine the oil saturation level. Uneven vibrations of the CH=CH− (cis) and CH=CH− (trans) groups can be observed in each 911 $cm^{-1}$ and 962 $cm^{-1}$ caused by the heating process. This process has the potential to convert cis-unsaturated fats to trans-unsaturated fats. It is proven that the increase in fat trans acids corresponds to a decrease in cis fatty acids (oleic fatty acids), as seen in Table 1. Monoglycerides, diglycerides, and triglycerides all contain ester bonds. Palm oil is almost entirely composed of triglycerides, with only a trace of mono-and diglycerides. Saturation ranges from 7% to 10% of total triglycerides, with palmitic fatty acids dominating. Unsaturated fatty acids are most commonly found in the sn-2 position of triglycerides. Under these conditions, more than 85% form ester bonds with glycerin, where the wavenumbers 1,032 $cm^{-1}$, 1,091 $cm^{-1}$, and 1,729 $cm^{-1}$ contribute to the interaction of C–O [29]. The bond indicates that the fatty acid remains bound to the glycerin, and the wavenumber of 2,974 $cm^{-1}$ contributes to the C-H bonds [16], which are most commonly found in hydrocarbons. Wavenumber 3,474.91$cm^{-1}$ indicates the interaction of C = O ester and triglycerides. Finally, detecting a wavenumber of 3,536 $cm^{-1}$ indicates secondary oxidizing compounds such as alcohols, aldehydes, and ketones [25, 30]. As the heating time is increased, the FFA value rises, and this reaction breaks the ester bond in the triglyceride, producing FFA and glycerol. Wavenumbers that meet the criteria are 722 $cm^{-1}$, 912.5 $cm^{-1}$, 966 $cm^{-1}$, 1,091 $cm^{-1}$, 1,418 $cm^{-1}$, and 1,654 $cm^{-1}$. The heated sample also shows a shoulder peak of about 1,700 $cm^{-1}$, corresponding to FFA production due to oxidation. The functional groups most affected by the oxidation reaction are carbonyl and alkene. The change can be attributed to band overlap (∼1,728 $cm^{-1}$) for forming aldehyde functional groups from oxidation and production of secondary products [32]. This observation is consistent with the data obtained for peroxide


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number for the mixture as shown in Figure 3. Fourier Transform Infra-Red (FTIR) spectroscopic measurements revealed functional groups = C - H (trans and cis), C - O esters, and double bonds = $CH_2$ that are susceptible to oxidation reactions, according to Lerma-Garcia *et al.* [29]. According to Russin [33], wave numbers 3,444 $cm^{-1}$, 2,854 $cm^{-1}$, and 1,100-1,270 $cm^{-1}$ correlate with PV use areas. In contrast, Guillen and Cabo (2002) selected wavenumbers 1,746 $cm^{-1}$, 1,728 $cm^{-1}$, 1,163 $cm^{-1}$, and 1,118 $cm^{-1}$ instead [25].

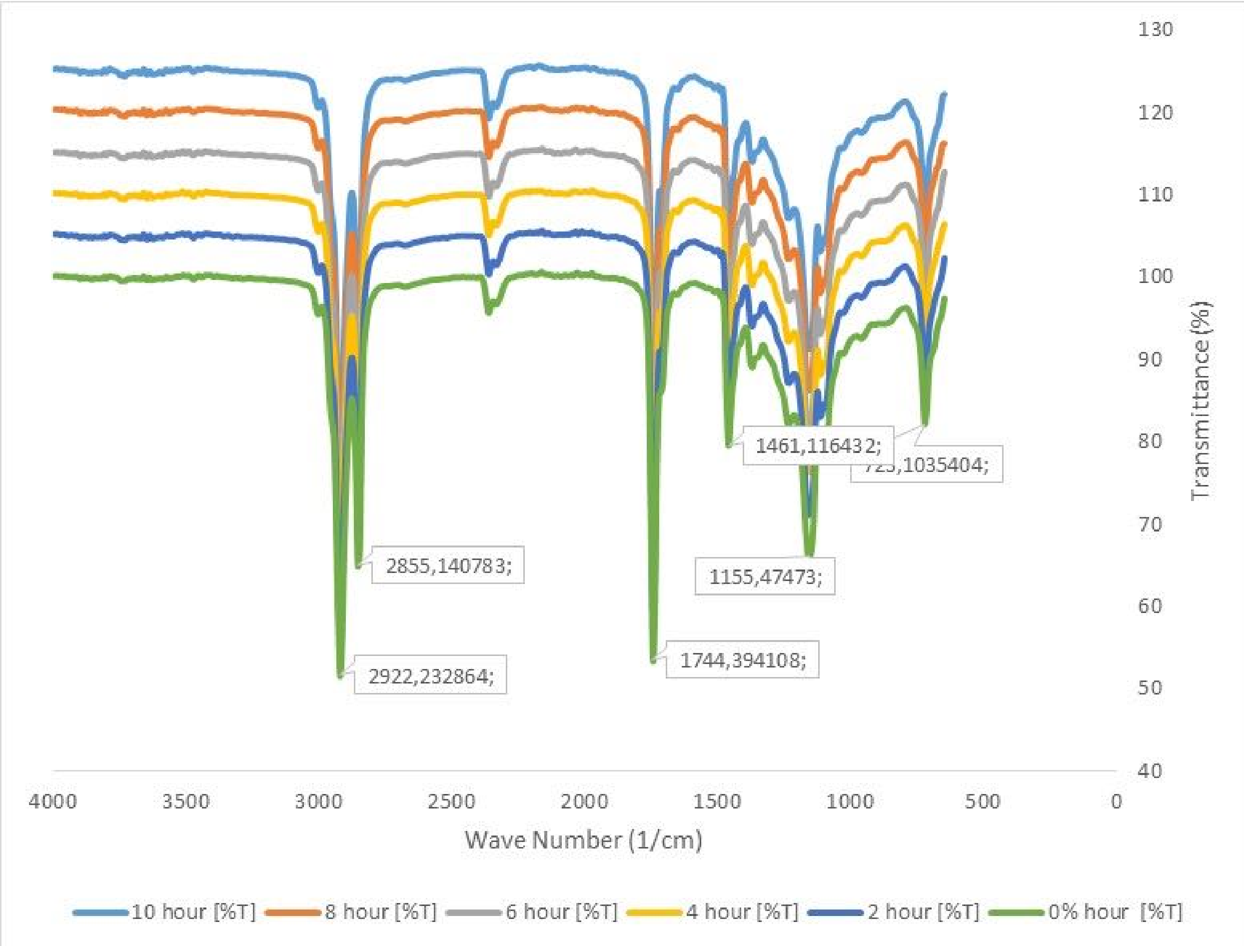


**Figure 4: FTIR spectra due to the prolonged heating effect of red palm oil**

## Fatty Acid Composition Using Gas Chromatography

As shown in Table 1, the heating process reduced the relative percentage of unsaturated fatty acids. The type of oil also influences the changes and stability during heat treatment due to differences in the fatty acid profile. Choe *et al.* [34], for example, found a significant decrease in linolenic, polyunsaturated and monounsaturated acids, with a relative increase in caprylic, palmitic, stearic and arachidic oleic acids. Red palm oil contains 42.22% and 10.02% oleic and linoleic acids, respectively, with a total unsaturated fatty acid content of 53.81%. Palmitic acids account for the majority of saturated fatty acids (40.27%), and a study that looked at changes in soybean oil and hydrogenated vegetable fat discovered a slight increase in the percentage of saturated fatty acids [20]. Others had discovered that the level increased as the heating time increased [21]. Polyunsaturated fatty acids oxidize more easily than saturated fatty acids. In general, the percentage of linoleic acid decreases with heating time, and in this study, the decrease for 10 hours was around 3.23%.

According to the literature, these losses are primarily caused by oxidation and polymerization reactions. According to double bond stereoisomerism, unsaturated fatty acids that can be cis or trans increase the formation of trans unsaturated stereoisomers when heated for 10 hours [8], and the increase was around 0.35%. Saturated Fatty Acids (SFA) and Monounsaturated Fatty Acids (MUFA) are more stable than unsaturated and Polyunsaturated Fatty Acids (PUFA) fatty acids. Long-term heating increased SFA content [9], and heating-related factors influenced monounsaturated fatty acid levels. The content of trans fatty acids increases with little or no prolonged heating, while essential fatty acids decrease by about 3.3%. Even though the content of saturated fatty acids decreases over time, the amount varies and, in some cases, increases.

The availability of essential linoleic and -linolenic acids, which are precursors to arachidonic, eicosapentaenoic, and docosahexaenoic acids, was also influenced by compounds formed during the breakdown of unsaturated fatty acids. For example, after 8 hours of heating at 180°C, the oleic content of red palm oil increased by 0.33%. Cella *et al.* [35] discovered a 0.2598% to 0.3491% increase in oleic acid content in cooking oil samples used for 6 hours 35 minutes to 10 hours 55 minutes.

Deep frying alters the physicochemical, nutritional and sensory properties of the oil. Many complex reactions, such as hydrolysis, isomerization, cyclization, oxidation and polymerization, occur during this process, altering the taste and decomposing the oil compounds [35]. Fatty acid oxidized products have taste and odor, and high levels in finished oils are undesirable because they can cause bad taste and shorten shelf life. Because of the accumulation of toxic substances in cooking oils and products, deteriorating cooking oil can have negative health effects on the human system. After repeated frying, the oil may degrade to the point where it is no longer fit for use and should be discarded.

## CONCLUSION, AND RECOMMENDATIONS FOR DEVELOPMENT

Increasing the heating time at 180°C can increase the peroxide value and acid number but decrease the iodine number. Red palm oil purchased online still meets the Indonesian National Standard for peroxide value but not for acid number. The high value of the Iodine number is due to the double-bonded carotene of red palm oil. Heating at 180°C only slightly changes the oil composition from unsaturated to saturated.

## CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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**Table 1: Effect of heating time on changes in the fatty acid composition of red palm oil**

| No | Fatty Acid (%) | Heating Time (Hour) | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0 | 2 | 4 | 6 | 8 | 10 |
| 1 | Linolenic Acid | 0.2276 | 0.2574 | 0.2548 | 0.2509 | 0.25 | 0.2434 |
| 2 | C 18:2 W6C (C-Linoleic Acid) | 10.8229 | 10.7317 | 10.6555 | 10.6431 | 10.5767 | 10.479 |
| 3 | C 18:1 W9C (C-Oleic Acid) | 42.2286 | 42.3299 | 42.3297 | 42.3315 | 42.3602 | 42.2399 |
| 4 | C 17:1 (Heptadecanoic Acid) | 0.0324 | 0.0261 | 0.024 | 0.0271 | 0.0259 | 0.0294 |
| 5 | C 16:1 (Palmitoleic Acid) | 0.241 | 0.1862 | 0.1981 | 0.1951 | 0.1915 | 0.1725 |
| 6 | C 8:0 (Caprylic Acid) | 0.0129 | 0.0162 | 0.0176 | 0.0171 | 0.0218 | 0.0208 |
| 7 | Omega 6 Fatty Acids | 10.8229 | 10.7317 | 10.6555 | 10.6431 | 10.5767 | 10.479 |
| 8 | Omega 3 Fatty Acids | 0.2276 | 0.2574 | 0.2548 | 0.2509 | 0.25 | 0.2434 |
| 9 | C 18:3 W3 (Linolenic Acid / W3) | 0.2276 | 0.2574 | 0.2548 | 0.2509 | 0.25 | 0.2434 |
| 10 | Polyunsaturated Fat | 11.1859 | 11.0852 | 11.0096 | 10.9678 | 10.8979 | 10.8131 |
| 11 | C 6:0 (Caporic Acid) | | 0.0123 | 0.0135 | 0.0211 | 0.0415 | 0.0105 |
| 12 | C 18:0 (Stearic Acid) | 4.1772 | 4.1758 | 4.1929 | 4.1975 | 4.2039 | 4.1817 |
| 13 | C 22:2 (Docosadinoic Acid) | 0.1354 | 0.0962 | 0.0993 | 0.0738 | 0.0712 | 0.0908 |
| 14 | C 17:0 (Heptadecanoic Acid | 0.0917 | 0.0914 | 0.0919 | 0.0906 | 0.0901 | 0.0932 |
| 15 | C 16:0 (Palmitic Acid) | 40.279 | 40.3375 | 40.3765 | 40.4096 | 40.4497 | 40.4024 |
| 16 | Unsaturated Fat | 53.8155 | 53.7572 | 53.6883 | 53.6489 | 53.595 | 53.3767 |
| 17 | Omega 9 Fatty Acids | 42.2286 | 42.3299 | 42.3297 | 42.3315 | 42.3602 | 42.2399 |
| 18 | C 15:0 (Pentadecanoic Acid) | 0.0417 | 0.0403 | 0.0414 | 0.04 | 0.0412 | 0.0414 |
| 19 | C 14:0 (Myristic Acid) | 0.9324 | 0.9357 | 0.9315 | 0.9306 | 0.9304 | 0.9203 |
| 20 | C 12:0 (Lauraic Acid) | 0.1618 | 0.1696 | 0.1714 | 0.1647 | 0.1619 | 0.1671 |


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|---|---|---|---|---|---|---|---|
| 21 | C 20:1 (Eicocyanic Acid) | 0.1276 | 0.1298 | 0.1269 | 0.1274 | 0.1195 | 0.1218 |
| 22 | Monounsaturated Fa | 42.6297 | 42.672 | 42.6787 | 42.6811 | 42.6971 | 42.5636 |
| 23 | C 10:0 (Capric Acid) | 0.0145 | 0.0151 | 0.0148 | 0.0151 | 0.0135 | 0.0147 |
| 24 | C 20:0 (Arachidic Acid) | 0.3483 | 0.3524 | 0.3506 | 0.3574 | 0.3561 | 0.3563 |
| 25 | Saturated Fat | 46.0595 | 46.1553 | 46.2142 | 46.2436 | 46.31 | 46.2083 |



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