

# Thermal degradation of coconut oil with free fatty acid, peroxide value, and moisture content indicators

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## ABSTRACT

Coconut oil is consumed by people for cooking purposes. It is usually subjected to long heating at high temperatures and often reheated and reused. There is a need to investigate the degradation of coconut oil without a food matrix to determine its inherent degradation properties if it is subjected to long thermal stress. This study investigates the thermal degradation of refined, bleached, and deodorized coconut oil (RBDCO). The RBDCO samples were heated at three different temperatures (150, 175, and 200 °C) for 12 hours continuously. Samples were collected every hour interval and analyzed for free fatty acid (FFA), peroxide value (PV), and moisture contents (MC). Results reveal that increased temperature and heating time lead to higher FFA, PV, and MC, indicating significant oil degradation. The findings suggest that maintaining optimal processing conditions is crucial for preserving oil quality and safety standards.

**Keywords:** vegetable oil, oil degradation, coconut oil

## INTRODUCTION

Coconut (*Cocos nucifera*) is one of the essential crops in tropical regions. The oil extracted from its fresh meat or kernel is economically important and used extensively in the culinary, pharmaceutical, and cosmetic industries (Santos et al., 2011). The major component of coconut oil is saturated fat (92%) of which 60-70% is made of medium-chain fatty acids (MCFAs) (Ferreira et al., 2019; Gervajio et al., 2020). MCFAs are lipids that are easily oxidized and are not retained in

the adipose tissue as opposed to long-chain fatty acids (LCFAs) (Liau et al., 2011). MCFAs have been linked to positive health benefits enhancing metabolism that helps lose weight (Huang et al., 2011; Nagao & Yanagita, 2010; Schönfeld & Wojtczak, 2016; Wallace, 2019), and lowering the levels of low- and very-low-density lipoproteins in the blood (Famurewa et al., 2018; Nevin & Rajamohan, 2004). Despite various issues and controversies (Dayrit, 2017b, 2017a; Sacks et al., 2017), coconut oil has emerged as the oil of choice for health-conscious individuals due to the surge in demand for plant-based oil choices. Aside from the health benefits it offers, the edible coconut oil offers rich flavor, distinct coconut aroma, and unique thermal properties.

Coconut oil has two (2) main variants: virgin coconut oil (VCO) and refined, bleached, and deodorized coconut oil (RBDCO). VCO is cold pressed from fresh mature coconut meat and is processed only by physical or natural methods without heat or chemicals (Dayrit et al., 2008; Santos et al., 2011). On the other hand, RBDCO is manufactured by extracting oil from dried copra and subjecting it to further refining, bleaching, and deodorizing processes (Gunstone, 2011). RBDCO is a consumer good that is sold commercially as an edible or cooking oil for frying purposes.

Deep-frying is one of the oldest and most common methods of preparing home-made food and in the food service industry due to its ability to enhance the sensory properties of various dishes affording distinctive taste and fragrance (Bordin et al., 2013; Liu et al., 2019). During the frying process, the oil is continuously heated at high temperatures of 150 to 190 °C in the presence of air and moisture (Choe & Min, 2007; Sharoba & Ramadan, 2012). As this happens, the frying oil undergoes physical and chemical changes, affecting its frying performance (Bordin et al., 2013; Gertz & Matthäus, 2008; Khan et al., 2011; Liu et al., 2019; Sharoba & Ramadan, 2012). Frying in hot oil results in a multitude of complex chemical reactions leading to the formation of various breakdown products. As the chemical reactions progress, the fat/oil's functional, sensory, and nutritional attributes will degrade to an extent where it becomes unsuitable for consumption necessitating its disposal (Sharoba & Ramadan, 2012). To ensure optimal performance, the frying oil must exhibit a sufficient level of thermo-oxidative resistance when subjected to prolonged high-temperature heating (Mba et al., 2016). The process of monitoring frying fats and oils entails the utilization of chemical indicators such as free fatty acid (%FFA), peroxide value (PV), and moisture content (MC). In the context of food quality, physicochemical analysis is of utmost importance. When coconut oil is subjected to heat during cooking or processing, especially in the presence of the food matrix, it undergoes thermal degradation, resulting in changes to its chemical composition and physical properties. Thorough studies that describe the degradation of coconut oil at high thermal stress without food matrix are not available, hence this work. By conducting a physicochemical analysis of the coconut oil alone, researchers can gain valuable insights into the extent and nature of these changes. In this study, physicochemical properties such as free fatty acid, peroxide value, and moisture content of bare coconut oil at high thermal cycles were analyzed. Understanding these factors can help ensure the safety and quality of the oil in terms of the potential formation of harmful compounds or their degradation products. Moreover, understanding the physicochemical properties of heated coconut oil allows the development of strategies to improve its properties and optimize processing conditions.

## METHODS

**Oil Samples.** The RBDCO sample was purchased from Frymax, a reliable Philippine manufacturer certified to conform to the rigid standards of the Philippine Food and Drug Administration.

**Chemicals.** Chemicals such as ethanol, sodium hydroxide, phenolphthalein were purchased from Yana Chemodities and Alyson's Chemical Enterprises and used as received.

**Physicochemical Investigation on the Degradation of Heated RBDCO.** The heating of RBDCO was carried out using an electric fryer equipped with a thermostat. The temperature of the oil was monitored using a thermometer. The oil was heated at three (3) different temperatures (150, 175, and 200 °C), which are close to the 180 °C home-frying temperature (Cordella et al., 2012), for 12 continuous hours. The sampling schedule was every hour resulting in a total of 36 samples analyzed. All treated samples were packed in amber bottles and stored at a refrigerated temperature of 4-5 °C until analyzed. All data obtained by physicochemical analyses (FFA, peroxide value, and moisture content) were tabulated and performed in triplicates.

**Analysis of Free Fatty Acid (FFA) in Heated RBDCO.** The free fatty acids were determined based on the Official Methods of Analysis of AOAC International (2012) using a titrimetric method. In this method, about 56 g of the oil sample was mixed with 50 ml of 95% ethanol. The solution was heated while thoroughly shaking the flask for complete homogenization. The solution was titrated with aqueous sodium hydroxide (0.1 N) until the color turned light pink. The amount of FFA was calculated as a percentage of lauric acid.

**Analysis of Peroxide Value (PV) in Heated RBDCO.** The PV analysis was performed at the Standard and Testing Division of the Industrial Technology Development Institute of the Department of Science and Technology. The peroxide value of the oil samples was determined by the titration method based on the Official Methods of Analysis of AOAC International (2012). Five (5.0) grams of oil was placed into a 250 ml Erlenmeyer flask. Acetic acid chloroform solution (three parts of glacial acetic acid and two parts of chloroform) was then added, and the solution was swirled vigorously until dissolved. Potassium iodide (KI) solution was added and allowed to stand for 1 minute with occasional shaking. Water (30ml) was added, and the solution was titrated with 0.1M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (sodium thiosulfate) under vigorous swirling until the yellow color almost disappeared. Starch solution (1%; 0.5ml) was added and the titration was continued until the blue color almost disappeared. The peroxide values of the oil samples were calculated using the following formula:

$$\text{Peroxide Value} \left( \frac{\text{meq}}{\text{kg}} \right) = \frac{S \times N}{W(g)} \times 100$$

Where S = amount of titration sample (mL); N = Normality of the sodium thiosulphate (meq/kg); W = weight of the oil sample (g)

**Analysis of Moisture Content in Heated RBDCO.** The method for the determination of the moisture content of heated RBDCO samples used the vacuum oven method (Official Methods of Analysis of AOAC International, 2012). This procedure includes weighing oil (3.0g) into tared aluminum moisture dishes, followed by drying in a vacuum oven for 16 hours. The oven temperature was set at 105 °C and a vacuum of <100 millibars. The moisture content was calculated using the equation:

$$\%MC_{wb} = \frac{\text{Wt of sample before drying} - \text{Wt of sample after drying}}{\text{Wt of sample before drying}} \times 100$$

## RESULTS AND DISCUSSION

**Free Fatty Acids (FFA) of Thermally Treated Coconut Oil.** Researchers and cooks have always shown interest in exploring the maximum lifespan of oils in deep-fat frying, especially for long frying and re-use purposes (Mba et al., 2016). The quality assessment of edible oil is largely influenced by the %FFA content. The raw %FFA in freshly prepared oils is generally low during production, especially after the refining process. The freshly opened RBDCO used in this current work, the initial value of %FFA as reported by the supplier is 0.027% (Table 1), which is within

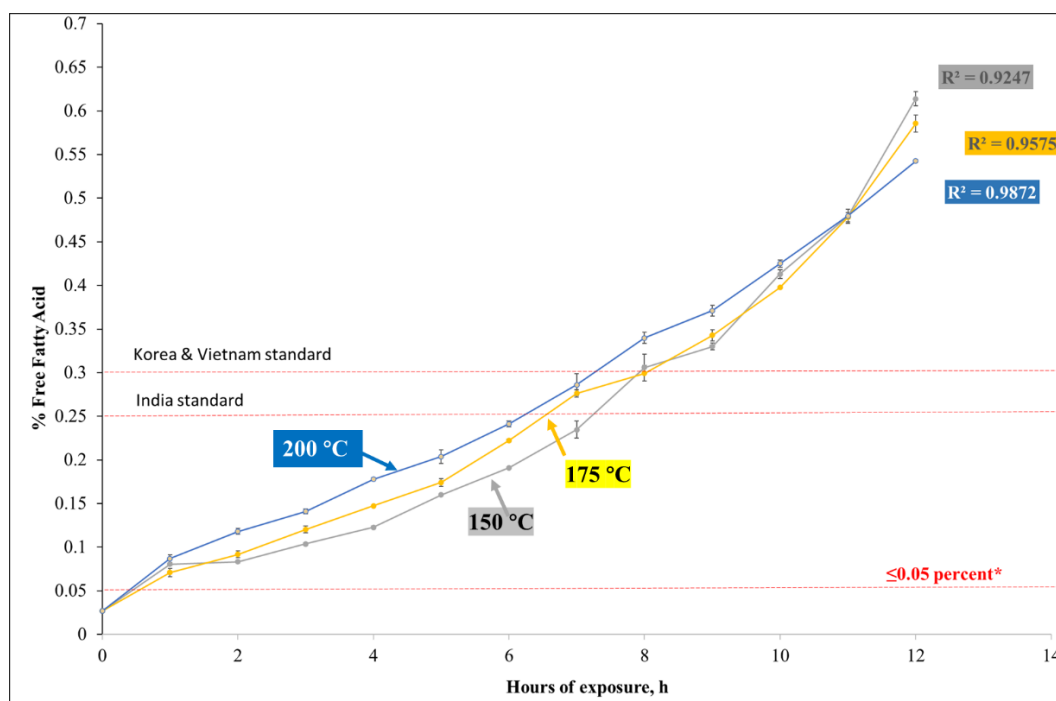
the acceptable standard of 0.05%. FFAs are undesirable in edible oils due to their negative effects on the oxidative stability, acidity levels, and flavor profile of the final product (Dunford, 2016). According to the voluntary industry guideline, the FFA content of refined edible oil must not surpass 0.05%, based on the weight of the oil. Within the food industry, frying oils with a free fatty acid content exceeding 2% are often discarded or supplemented with fresh oil to reduce the FFA concentration. In this work, the %FFA of RBDCO was monitored to assess the quality of coconut oil following exposure to varying temperatures and various heating times.

**Table 1. Physicochemical properties of coconut oil sample before thermal stressing**

Physicochemical Properties	Method	Observations	Standard
Physical Appearance	Visual	Light yellow liquid at room temp	Light yellow liquid at room temp
Taste/Odor	AOCS Cg 2-83	Creamy	Creamy to bland
Extraneous matter	Filtration (Whatman #541)	Free from foreign matters	Free from foreign matters
%FFA (as Lauric acid)	AOCS Ca-40	0.027	0.05 max
Peroxide value, meq/kg	AOCS Cd 8-53	0.237	1.0 max
Moisture	AOCS Ca 2e-84	0.038	0.1 max

*Note:* The method and analysis values are provided by the supplier

Figure 1 shows the changes in the %FFA of RBDCO subjected to varying combinations of temperature and heating time. The observed trend indicates a positive correlation between the concentration of FFA and the duration and temperature conditions. This is analogous to the research conducted by Mba, et al. (2016) employing canola oil and palm oil. Similar observations were made by Sharoba and Ramadan (2012) on the sunflower, cottonseed, and palm olein oils used for deep frying, with varying durations of exposure. The process of oil oxidation and chemical changes during heating is marked by an increase in the concentration of free fatty acids and a reduction in overall unsaturation levels (Nduka et al., 2021). The fatty acid, being the primary constituent of the oil, exerts an impact on its oxidative stability. At temperatures ranging from 150 to 200 °C, it has been observed that the rise in FFA levels is directly proportional to the duration of heating. The primary oxidative process in oils, involving the hydrolysis of triacylglycerols and the decomposition of hydroperoxides, is responsible for the observed elevation in FFA during successive deep-frying cycles (Dodoo et al., 2022). An increase in FFA following the process of deep-frying indicates that the oils used are prone to instability and have a high likelihood of undergoing rapid oxidation, resulting in the formation of potentially harmful oxidized compounds. This oxidation process has the potential to negatively impact the overall quality of the fried food (Dodoo et al., 2022).



The voluntary industry standard for FFA content in refined edible oil is  $\leq 0.05$  percent

Figure 1. Transformation of free fatty acid levels (%FFA) of heated coconut oil at various temperatures and time exposures

It is evident from Figure 1 that there is a distinct and consistent rise in FFA concentrations at temperatures of 150, 175, and 200 °C. The linear relationship was confirmed by the high R-squared values of 0.9247, 0.9575, and 0.9872, respectively. These data points demonstrate a significant positive correlation between the duration of thermal treatment and the concentration of free fatty acids in the oil. The results indicate that as the duration of exposure to these specific temperature ranges increases, there is a consistent and predictable increase in the concentration of free fatty acids in the oil. Another noteworthy finding is the reversal of FFA levels between 150 °C and the elevated temperatures (175 and 200 °C) at the 12<sup>th</sup> hour. This could potentially be attributed to reaction fatigue. Heating has the potential to cause alterations in both the composition and structure of coconut oil. At a temperature of 200 °C, the oil likely underwent extensive chemical transformations over time, potentially resulting in a decrease in the presence of substances required for the production of FFAs, such as reactants or catalysts. On the other hand, the oil subjected to a temperature of 150 °C might have undergone a less severe conversion, thereby enabling the reaction to persist and yield elevated levels of free fatty acids (FFA) after the 12<sup>th</sup> hour. However, to ascertain the precise underlying mechanisms, additional research would be required to examine the specific chemical reactions, reaction kinetics, and compositional alterations that take place during the thermal treatment. It is noteworthy to mention that all temperatures and duration combinations tested did not yield free fatty acid levels surpassing the 2% threshold set by food manufacturers for used oil. This suggests that the coconut oil subjected to thermal treatment maintained its composition within the acceptable parameters as defined by the industry standard.

It is crucial to acknowledge that although temperature and duration are influential factors in the generation of FFA, there are additional variables that can contribute to the levels of FFAs in coconut oil. The composition of food, which includes different types of fatty acids and triglycerides present, can influence the rate at which FFAs are generated during heat processing. Typically, the elevation in FFA concentrations is attributed to the cleavage and oxidation of unsaturated fatty acids, leading to the formation of carbonyl compounds. These carbonyl compounds subsequently undergo oxidation, resulting in the production of low molecular weight fatty acids during frying processes (Sharoba & Ramadan, 2012). Moreover, the elevation in FFA

levels is more pronounced in the presence of heated food, as the composition of the food significantly influences the generation of FFA. In the research conducted by Sharoba & Ramadan (2012), it was observed that the water derived from fried potato facilitated the process of hydrolysis cleavage of the oil. Similar results were observed in the experiment conducted where Sorghum bug oil was utilized for frying pre-fried potatoes at a temperature of 175°C (Mariod et al., 2006). The moisture release by the potato is believed to have caused the FFA to exceed 2%. This is analogous to the investigation conducted by Mba et al (2016) on the analysis of processed canola oil. It has been determined that refined canola oil, despite initially having low FFA levels, demonstrated the most significant degradation of FFA.

The codex Alimentarius is a collection of international food standards, guidelines, and codes of practice established by the Food and Agriculture Organization (FAO) and the World Health Organization (WHO). Table 2 shows the maximum allowable FFA limits in oils. The Codex, however, did not specify any maximum limits. Various countries have their own specific requirements and regulations regarding FFA. These requirements are typically established by national regulatory bodies or industry standards organizations. In China, a high maximum limit of 1.5% has been set. In India, South Korea, and Vietnam, the %FFA is in the region of 0.25 to 0.30%. The Philippines and Thailand, however, did not specify the maximum limits. It might be high time to review this and influence the country to set the maximum limits.

**Table 2. National standards and requirement of %FFA of refined vegetable oil**

Country	%FFA (maximum allowable limit)
Codex	-
China	1.50
India	0.25
South Korea	0.30
Thailand	-
Vietnam	0.30
Philippines	-

This study enhanced the current understanding of the impact of temperature and time exposure on the levels of FFA in thermally processed coconut oil. The results offer valuable insights for professionals working in the food processing sector, empowering them to make well-informed choices regarding the most favorable processing conditions and quality control measures. Moreover, this study highlights the significance of complying with industry norms, as the examined temperature and duration combinations consistently stayed within the permissible limits of FFA levels for refined coconut oil.

**Peroxide Value (PV) of Thermally Treated Coconut Oil.** During the frying process, a series of chemical reactions occur, including oxidation, hydrolysis, polymerization, and fission (Waghmare et al., 2018). Free radicals are responsible for initiating the process of oxidation that occurs during the frying process. The primary variables that impact oxidation include frying temperature, food material composition, moisture levels, enzymatic reactions, light exposure, and the presence of catalysts. Triglycerides undergo oxidation in one of their unsaturated fatty acyl groups.

The quality of oils is determined by their chemical compositions, specifically the percentage of unsaturation. The PV is a quantitative measurement that assesses the degree of primary oxidation in oils, specifically the process of rancidification. This value is influenced by factors such as temperature, time, and light exposure. The process of oil rancidity can lead to the formation of potentially harmful compounds that are linked to adverse long-term health effects, including obesity, diabetes, neurological disorders, cardiovascular issues, and cancer (Bustani & Soni, 2023; Kaleem et al., 2015).

The PV is a commonly employed technique for assessing the initial oxidation of oils. Hydroperoxides are the initial outcomes of lipid oxidation, commonly known as peroxides. Peroxides are a class of organic compounds that are derived from triglycerides. Hydroperoxides undergo a chemical reaction with iodide ions, resulting in the formation of iodine. This iodine can then be quantitatively determined by conducting a titration using thiosulfate. An oil with a peroxide value exceeding 10 meq/kg is deemed to be rancid, rendering it unsuitable for consumption as a food product. A peroxide value below 2 meq/kg is considered to be within acceptable limits for fresh oils (Waghmare et al., 2018).

In this work, peroxide values of coconut oil under different temperature-time conditions were investigated (Table 3). The initial peroxide value of the coconut oil sample was 0.237 meq/kg, and the maximum acceptable value for refined oil is 1 meq/kg (Table 1). From Table 3, it can be observed that peroxide values increased with increasing temperature and heat cycle. The same is observed in the study of algal, sunflower, and palm oils, wherein peroxide values increase as the frying cycle is increased (Waghmare et al., 2018). It can also be noted that all temperature variables exceeded the peroxide value of 1 meq/kg within the first hour. This indicates that oxidative rancidity, a sign of oil degradation, was initiated early in all samples. The higher temperatures led to accelerated oxidation compared to lower temperatures. The sample at 150 °C reached a peroxide value of 10 meq/kg by the third hour. This threshold signifies that the oil became rancid and unsuitable for food use at that temperature. Similarly, the 175 °C heated sample exhibited high peroxide values right from the first hour indicating immediate rancidity. In a study using palm oil for deep frying of potatoes, a PV of 10 meq/kg was already obtained at 20 minutes of frying time (Xu et al., 2015). Thus, it can be concluded that food plays a significant role as a contributing factor in the formation of hydroperoxides in frying oil.

Interestingly, for samples heated at 150, 175, and 200 °C, the peroxide values started to decrease after about the 8th hour. This decline might be due to the depletion of initial reactants or a shift in the balance between oxidation and degradation reactions. The same observation was made in a study of crude palm oil subjected to heating (Fabien et al., 2014) where the PV increased at high temperatures (from 120 °C) at the beginning of heating and decreased after a certain heating time. In summary, the findings of this study highlight the significant impact of time and temperature on peroxide values and the progression of oxidative rancidity in oils. The study emphasizes the importance of considering temperature conditions when storing and using oils in the food industry. Further investigations could explore the underlying chemical reactions and mechanisms to enhance our understanding of oil oxidation and develop effective preservation strategies.

**Table 3. Peroxide values of coconut oil at different time-temperature combinations**

Time, hr	Temperature, °C		
	150	175	200
1	1.542 ± 0.057	11.131 ± 0.021	8.087 ± 0.008
2	2.380 ± 0.001	16.174 ± 0.013	15.170 ± 0.005
3	10.347 ± 0.006	36.401 ± 0.037	17.162 ± 0.021
4	26.255 ± 0.205	40.765 ± 0.544	24.302 ± 0.020
5	39.751 ± 0.367	38.409 ± 0.319	23.239 ± 0.062
6	43.441 ± 0.794	36.415 ± 0.051	21.169 ± 0.104
7	50.322 ± 2.871	39.487 ± 0.054	17.187 ± 0.034
8	72.027 ± 1.605	37.410 ± 0.005	20.186 ± 0.069
9	65.569 ± 0.681	32.315 ± 0.073	16.107 ± 0.090
10	53.564 ± 1.611	31.374 ± 0.043	16.164 ± 0.034
11	52.718 ± 1.225	34.395 ± 0.033	12.110 ± 0.028
12	47.548 ± 0.533	32.411 ± 0.018	12.149 ± 0.017

**Moisture Content (MC) of Heated Coconut Oil.** Excessive moisture in edible oil can lead to quality deterioration and reduced shelf life. Moisture provides a favorable environment for the growth of microorganisms such as bacteria, yeast, and molds, which can spoil the oil and make it unsafe for consumption. These microorganisms can cause off-flavor, odors, and even produce toxins, compromising the quality and safety of the oil. Moisture content in edible oil can promote oxidative reactions, leading to rancidity. Water acts as a catalyst in the oxidation process, accelerating the breakdown of fats and oils. Oxidative rancidity results in the formation of off-flavors, and off-odors, and a decrease in the nutritional value of the oil (Negash et al., 2019). Controlling moisture levels helps maintain the oxidative stability of edible oil and prolong its shelf life. Excessive moisture in the frying oil can lead to increased foaming, splattering, and poor frying performance. The presence of water in the oil can cause steam explosions, resulting in oil splashes that can cause burns and pose safety risks in the kitchen.

The moisture content affects the quality and texture of fried food products. Excess moisture in the frying oil can lead to greasy, soggy, and less crispy fried foods. Proper moisture control in the oil helps achieve the desired texture, crispiness, and overall quality of fried food items. The thermal hydrolysis process of oils is triggered by the application of heat, air, and moisture. This combination of factors initiates a thermochemical reaction that ultimately results in the degradation of the oil. The moisture content serves as an indirect indicator of the oil's quality and its ability to withstand thermal oxidation. Figure 2 shows the changes that took place in coconut oil after subjecting to heat at various temperatures and time combinations. According to the WHO/FAO guideline, the maximum permissible limit for moisture content in edible oils is set at 2% (Negash et al., 2019). In commercial frying operations, the moisture content in edible oil is carefully controlled. When the moisture content of edible oils falls within the range of 0.05 to 0.3%, it indicates a high likelihood of rancidity development (Negash et al., 2019).

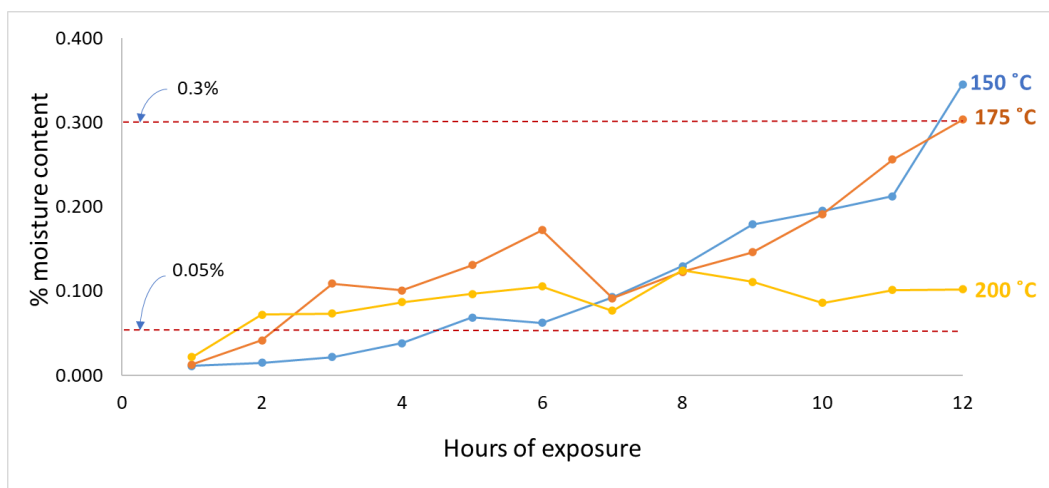


Figure 2. Moisture content of heated coconut oil at different temperatures and time combinations

Results show the moisture content of coconut oil increased with an increase in time exposure at all temperatures. This finding suggests that the heating process caused the oil to absorb moisture from the surrounding environment. As the exposure time prolonged, more moisture was absorbed, leading to higher moisture content. This can be attributed to the headspace moisture migrating into the bulk oil like that observed in the study of moisture content of corn oil heated to 60, 100, and 140 °C (Park et al., 2014). Additionally, the study of Schiller et al (2002) presented that H<sub>2</sub>O is produced as oxygen inserts into the C-H bond of a saturated fatty acid leading to the production of ketone. Contrary to the study of Dodoo et al., (2022) where repeated heating of oil resulted in moisture loss, which they attributed to the evaporation of water in the frying medium as the temperature was raised. The results in this current work showed that as the temperature increased, there was a corresponding increase in the moisture content of the coconut oil. This trend was observed at all temperatures. The high temperatures likely accelerated the moisture



absorption process. The increased thermal energy at high temperatures might have facilitated the movement of moisture molecules, leading to enhanced moisture uptake. When considering the combined effects of temperature and time exposure, it was evident that high temperatures and long exposure times resulted in high moisture content. One thing is certain, the higher the moisture content, the more it is susceptible to thermal hydrolysis (Dodoo et al., 2022). Thus, it is crucial to control the moisture content during processing and storage to ensure product quality and safety.

## CONCLUSIONS

The thermal degradation of RBDCO showed that as the temperature and duration of exposure to heat increase, there is a corresponding increase in the concentration of free fatty acids, peroxide values, and moisture content in the oil. These changes indicate the chemical degradation processes that can lead to the formation of harmful compounds, compromising the quality of the oil. The study also highlights the importance of adhering to industry standards and guidelines regarding maximum allowable limits for FFA, PV, and MC in refined edible oil. It emphasizes the need for proper monitoring of physicochemical properties to ensure the safety and quality of oils used in food processing. Overall, the findings emphasize the need for caution when reusing and reheating coconut oil, especially at high temperatures, due to the risk of chemical degradation and potential health implications. These results contribute valuable insights into the field of edible oil degradation and call for a reassessment of national standards to ensure consumer safety.

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