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Effect of Hydrogenation Process in Several Palm Oil Derivatives on its Application as Edible Wax to Replace Paraffin in Wax Applications

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ABSTRACT

Wax is mostly produced using paraffin as its formulation, but nowadays, for food safety and environmental reasons, edible wax has become highly possible as a substitute. The hydrogenation process has been carried out on several derivatives of palm oil, namely Refined Palm Stearine (RPS), Refined Palm Oil (RPO), Palm Soft Stearin (SST), Palm mid-olein (MOL), and Palm Mid Fraction (PMF) through 3 types of hydrogenation processes, namely: (1) complete hydrogenation with a target iodine value <1, (2) partial hydrogenation with limiting hydrogen gas consumption, and (3) partial hydrogenation with limiting reaction time. The hydrogenated products were characterized and compared to commercial paraffin and wax according to several parameters such as Solid Fat Content, crystallization rate, melting rate, melting point, smoke point, and flash point. Furthermore, the hydrogenated products were applied to the wax products and a mixture of hydrogenated and paraffin products. The wax produced was then evaluated based on several parameters, such as hardness, combustion flame, melting diameter, and combustion time. The findings concluded that all hydrogenated products can be used as raw materials for wax, either their self or hydrogenated and paraffin formulation products. Waxes made from Refined Hydrogenated Palm Stearin (RHPS)-1 and Refined Hydrogenated Palm Oil (RHPO)-1 are the hardest wax in terms of texture. In contrast, in the case of flame, the waxes made from hydrogenated products formulated with paraffin and paraffin itself have brighter flame than the others and Refined Hydrogenated Soft Stearin (RHSST)-1 and Refined Hydrogenated Palm Mid Fraction (RHPMF)-1 waxes are the waxes that burn the longest. Each wax has certain characteristics that can be related to the hydrogenated product used.

Contribution to Sustainable Development Goals (SDGs):

SDG 3: Good Health and Well-being
SDG 9: Industry, Innovation, and Infrastructure
SDG 12: Responsible Consumption and Production
SDG 13: Climate Action

1. INTRODUCTION

1.1. Research Background

Wax is a type of non-polar fat with a long chain that has many uses in daily life. Natural wax is generally formed from the combination of fatty acid esters and long-chain alcohols, providing unique and beneficial properties. However, in recent years, the hydrogenation process has been the main focus in wax making, mainly to produce edible wax as an alternative to paraffin. Hydrogenation is a chemical process where hydrogen is added to fats or oils to produce a product that is more saturated, stable, and safe for consumption [1].

Adding hydrogenation to wax-making using edible wax involves several important steps. First, raw materials, such as vegetable oil, are hydrogenated to increase the melting point and stability. Then, hydrogenated oils are mixed with additional materials, such as stearic acid, to increase the texture and durability of the wax. Edible wax resulting from this process is not only environmentally friendly but also safe to use in applications that have direct contact with food, making it a healthier choice compared to petroleum-derived paraffin. The results of the study showed that waxes 1.5 cm in diameter, 17 cm in length, and weighing 30 grams [2] from hydrogenated edible wax can burn for five hours. This reflects the good efficiency and durability of wax, similar to conventional paraffin wax. In this quality, edible wax not only functions as a light source but also provides safer and more environmentally friendly alternatives for various purposes, including emergency lighting and food decoration. The hydrogenation process used ensures that the wax is stable, not easily melted, and has excellent performance, providing a fun and safe experience for users.

Paraffin, as a basic ingredient in waxes, is a non-biodegradable petroleum by-product and has many problems, including not being edible, contact with products causing unsafe conditions when consumed, and when burned, it produces small particles, such as soot, that are not friendly to the environment and health. The smell produced from burning can have a negative effect on respiration [3]. However, the availability of paraffin is decreasing, considering the increasingly sophisticated technology of processing petroleum, thus reducing the presence of by-products. Therefore, many studies have been conducted to find alternatives to replace paraffin, such as Hydrogenated Palm Olein IV 64 [4], Hydrogenated Palm Stearin [5], hydrogenated soybean oil [6], Hydrogenated Rice Bean Oil, and several other vegetable oil derivatives. The hydrogenation process applied is also different for each type of oil. Characteristics of each raw material will really affect the quality of the final products to be formed and will really affect the quality of wax and edible wax produced.

The sustainable palm oil process to produce functional palm oil derivative products generally produces by-products that still have economic values, such as Soft Stearin, Palm Mid Olein, and Soft Palm Mid Fraction produced from the second fractionation of Refined Palm Olein, which still has limited application due to their characteristics and chemical properties. Therefore, this study aimed to utilize palm oil derivatives for potential applications, such as edible wax, to replace paraffin in candle applications.

1.2. *Research Objective*

This study aims to analyze the effect of several hydrogenation processes on the characteristics of the wax produced from several types of palm oil products.

2. MATERIALS AND METHODS

This study used a laboratory-scale experimental design with a comparative method. In this design, a comparison was carried out between 4 - 5 types of palm oil, which underwent a complete hydrogenation process with a target iodine number <1, hydrogenation with limited hydrogen gas, and hydrogenation with limited time. Thus, hydrogenated oil will be produced with different characteristics, and the results of the final product will be applied in laboratory-scale wax making.

2.1. *Materials*

In this study, the materials used were obtained from the Research and Development Laboratory of PT Multimas Nabati Asahan, which is in the form of various types of palm oil derivatives, such as Refined Palm Stearin (RPS), Refined Palm Oil (RPO), Palm Soft Stearin (SST), Palm mid Olein (MOL), and Palm Mid Fraction (PMF), Nickel Catalyst Pricat 9930 supplied by Johnson Matthey containing 22% of Nickel coated with hydrogenated fat, solid technical grade Paraffin Wax commonly available in chemical supply stores, Industrial Wax available in the market as a comparison, Technical Phosphoric Acid at a concentration of 85% and Crystalline Citric Acid, which were then dissolved in water at a concentration of 50%.

2.2. *Tools and Equipment*

A set of hydrogenation pilot plant, which was 1 reactor connected to a vacuum pump and electric heater (as shown in Figure 1) and laboratory glassware used for refining, blending, preparation, and other activities, as well as for wet chemical analysis equipment, including beakers, Erlenmeyer, measuring pipette, dropping pipette, three-necked round-bottom flask, distillation flask, cooling condensers. A set of pilot plant equipment (Figure 1) was used for the refining process. Gas chromatography equipment was used to analyze the characteristics of fatty acids. A penetrometer was used to measure the material hardness. Melting Point Apparatus was used to measure melting point. Nuclear Magnetic Resonance Apparatus was used to analyze the Solid Fat Content, crystallization rate, and melting rate. Flash Point Analyzer was used to measure flash points and smoke points.

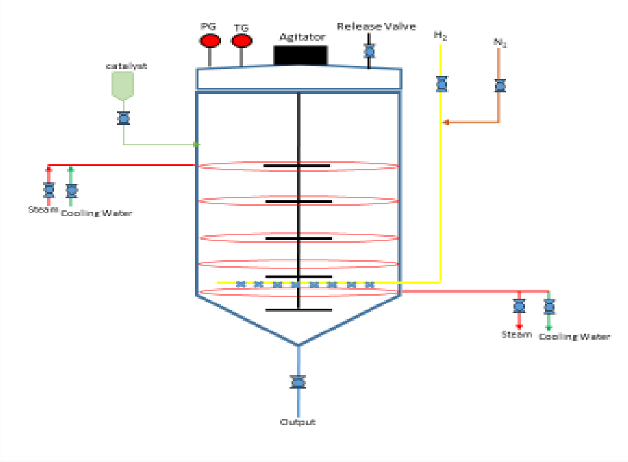

Figure 1 Schematic diagram of a pilot plant used for the hydrogenation process

2.3. *Hydrogenation and Refining Processes*

The hydrogenation process of each oil was carried out in a stirred and closed reactor at a pressure of 5.0 bar. 1500 gr of oil with nickel catalyst was put in the reactor, and nitrogen was transferred to remove oxygen in the reactor for several minutes, then heated slowly until the desired reaction temperature was reached, i.e. 150 - 190 ºC. Hydrogen gas was transferred gradually into the reactor until the amount of hydrogen gas consumed or reaction time met the desired reaction target. After the hydrogenation endpoint was reached, the supply of hydrogen gas was closed, and the heater was turned off. After that, the oil was cooled to 50 – 60 ºC. Then, the reactor was opened, and the filtrate produced was filtered to remove the remaining catalysts.

The hydrogenation process was carried out with 3 different treatments: (1) full hydrogenation, (2) partial hydrogenation with a limited amount of hydrogen gas, and (3) partial hydrogenation with limited time. In treatment 1, each palm oil derivative was hydrogenated until the product reached a maximum iodine number of 1. In treatment 2, the hydrogenation process was carried out with a limit on the amount of hydrogen used, which

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was 300 Nm³. Meanwhile, in treatment 3, the oil was hydrogenated with a fixed reaction time of 120 minutes.

Oil produced from the hydrogenation process was then purified. Hydrogenated oil purification was carried out with the following procedures: 1500 gr of raw materials in the vacuum flask was weighed, heated on the stirred heater in vacuum conditions to a temperature of 90 – 100 °C, and left for 1 hour for the drying process, and evaporation of water vapor. 0.04 - 0.05% of phosphoric acid was added at a concentration of 50% and left for 15 minutes. 0.030 - 0.045% of citric acid at a concentration of 85% was added, then the mixture was stirred for several minutes until mixed. 0.6% of bleaching earth was added at 0.6% of oil weight; then, it was stirred for 30 minutes for an optimum bleaching process. Then, the slurry was filtered using filter paper connected to the vacuum. The filtrate produced was then put into a deodorization glass flask.

All pilot plant equipment was then assembled and ensured that there were no leaks. Oil was heated in the heater up to 80 °C. Then, the vacuum pump was turned on, the vacuum valve that was directly connected to the deodorizer was opened, and sparging was ensured to function properly. Oil heating was continued until 250 °C for 4 hours, and sparging and vacuum were ensured to function properly. After the specified time, the heater was turned off, and the oil was left to cool naturally while maintaining sparging and vacuum to prevent further oxidation. The cooled oil was then analyzed and characterized following the required parameters, such as iodine number, solid fat content, melting point, smoke point, crystallization rate, fatty acid composition, and melting rate.

2.4. Candle Making

The application of hydrogenated oil as edible wax was carried out in the laboratory by limiting it to wax-making without adding coloring agents, fragrances, and other additives. The procedures were as follows: paraffin and hydrogenated oil were melted at 10 degrees above the melting point. The wick was cut according to the length of the wax mold, with an additional 5-10 cm, and inserted into the mold by clamping it with both hands. The wick was placed exactly in the center. Paraffin and hydrogenated oil were poured into the mold slowly and simultaneously. The mold was filled until the specified height. The wax was left to freeze naturally overnight. After 24 hours, the wax was removed from the mold and then left to cool again for at least 5 - 7 days in open air. Table 1 shows the product designation for each treatment.

3. RESULTS AND DISCUSSION

3.1. Hydrogenation and Product Produced

Figure 2 shows the photograph of oil to be used as raw materials for the hydrogenation process, and Figure 3 shows the hydrogenation products in flakes for wax application.

Figure 2. Photograph of palm oil derivatives used as raw materials for the hydrogenation process

Figure 3. Hydrogenation products in flakes form for use in wax application

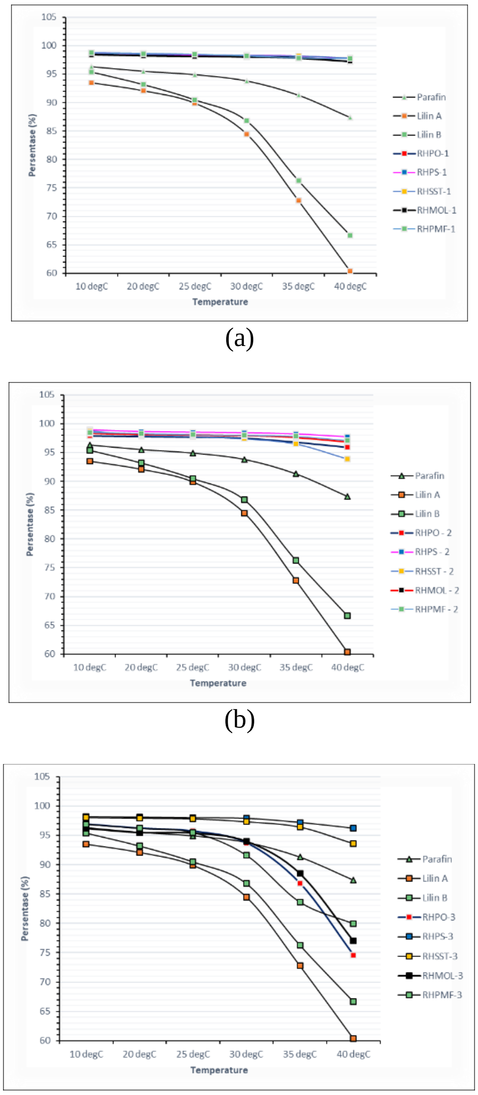
Table 1.: Hydrogenation process and the product produced

The hydrogenation process is carried out	**Raw Materials**	**Product Produced**
Fully Hydrogenation (Fully Hydrogenation) **Treatment 1**	RPS Refined Palm Stearin	RHPS - 1 Refined Hydrogenated Palm Stearin - 1
	RPO Refined Palm Oil	RHPO – 1 Refined Hydrogenated Palm Oil – 1
	SST Refined Palm Soft Stearin	RHSST – 1 Refined Hydrogenated Soft Stearin - 1
	MOL Refined Palm Mid Olein	RHMOL – 1 Refine Hydrogenated Palm Mid Olein – 1
	PMF Refined Palm Mid Fraction	RHPMF – 1 Refined Hydrogenated Palm mid Fraction - 1
Partial Hydrogenation with Limited Hydrogen gas (Partial Hydrogenation) **Treatment 2**	RPS Refined Palm Stearin	RHPS - 2 Refined Hydrogenated Palm Stearin - 2
	RPO Refined Palm Oil	RHPO – 2 Refined Hydrogenated Palm Oil – 2
	SST Refined Palm Soft Stearin	RHSST – 2 Refined Hydrogenated Soft Stearin - 2
	MOL Refined Palm Mid Olein	RHMOL – 2 Refine Hydrogenated Palm Mid Olein – 2
	PMF Refined Palm Mid Fraction	RHPMF – 2 Refined Hydrogenated Palm mid Fraction – 2
Partial Hydrogenation with limited time (Partial Hydrogenation) **Treatment 3**	RPS Refined Palm Stearin	RHPS - 3 Refined Hydrogenated Palm Stearin - 3
	RPO Refined Palm Oil	RHPO – 3 Refined Hydrogenated Palm Oil – 3
	SST Refined Palm Soft Stearin	RHSST – 3 Refined Hydrogenated Soft Stearin - 3
	MOL Refined Palm Mid Olein	RHMOL – 3 Refine Hydrogenated Palm Mid Olein – 3
	PMF Refined Palm Mid Fraction	RHPMF – 3 Refined Hydrogenated Palm mid Fraction - 3

a. Solid Fat Content (SFC)

SFC is used to determine the percentage of solid fractions in oil or fat at a certain temperature. It is usually measured at 10 – 40 degrees with an interval of 5 degrees. This parameter will show changes in fat consistency when temperature changes, and thus, it is required for wax application.

Figure 4 shows the SFC of hydrogenated products obtained from different treatments: fully hydrogenated process (treatment 1), partial hydrogenation with hydrogen gas consumption of 300 NM^3 (treatment 2), and partial hydrogenation with reaction time of 120 minutes (treatment 3). From Figure 4a, treatment 1 resulted in hydrogenated oils with similar characteristics. This shows that a fully hydrogenated process results in similar SFC characteristics for all types of oils. Furthermore, fully hydrogenated oils have harder characteristics compared to paraffin and wax.



(a)

(b)

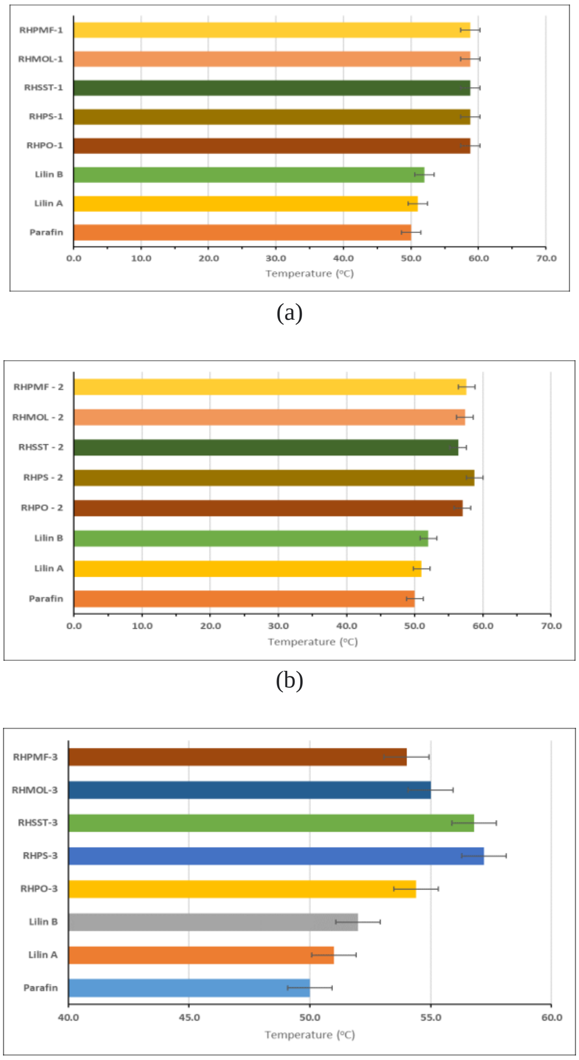
(c)

Figure 4 Solid Fat Content in hydrogenated product: (a) treatment 1, (b) treatment 2, (c) treatment 3

In treatment 2 (Figure 4b), the characteristic was almost similar to that in treatment 1, but it tended to be softer at higher temperatures. This could be an indication that hydrogenated oils with a limited amount of hydrogen will easily melt when applied as wax. In treatment 3 (Figure 4c), the time for the hydrogenation process was limited. Therefore, the double bonds in unsaturated compounds were not completely converted into saturated compounds. As a result, the wax produced will be wetter and not resistant to high temperatures.

b. Melting Point

The melting point is the temperature at which materials begin to melt, which affects the process and performance of wax. Figure 5 shows that the melting point of hydrogenated oils was in the standard range set by SNI 7351:2014 regarding the melting point characteristic of wax, which is 45 – 65 °C for paraffin wax and 62 – 65 °C for beeswax. According to Figure 5, the modified palm oil derivative products have higher melting points than paraffin. This indicates the stability of the products since products with higher melting points tend to be more stable at higher temperatures.



(a)

(b)

(c)

Figure 5. Melting points of hydrogenated product: (a) treatment 1, (b) treatment 2, (c) treatment 3

Treatment 1 resulted in hydrogenated oils with the highest melting point, followed by treatment 2. However, for treatment 3, the melting points varied depending on the types of oil. When compared to wax and paraffin, the hydrogenated oils have a higher melting point. This means that when applied to wax, it will produce a more durable wax. The melting point of wax is influenced by the melting point of the wax base used and by the concentration of active substances. If the concentration of active substances is high, the melting point of wax is low, and vice versa [7]. The lower the concentration of essential oil, the higher the melting point of wax [8].

c. Smoke Point and Flash Point

Smoke point and flash point indicate the temperature at which materials begin to melt and emit smoke before ignition occurs. Ideal wax should have a high smoke point so that it is not easily flammable and produces smoke when used. Flashpoint indicates the minimum temperature at which vapor from the burning substance can form a sufficient mixture with the air to start combustion.

Paraffin has a smoke point of 220°C, which is quite high to meet the requirements as raw materials for wax. However, wax from the market used in this study had a higher smoke point of 222°C, indicating that market wax contained materials that are not only paraffin but there were other compositions such as oil/fat and stearic acid, to increase the smoke point [9]. The more the paraffin composition in wax preparation, the more solid the wax produced [10].

Figure 6 shows the smoke point and flash point of hydrogenated oils from all the treatments. The results showed that the smoke point and flash point of almost all hydrogenated oils had similar results. This means that all hydrogenated oils have the potential to replace paraffin in wax applications.

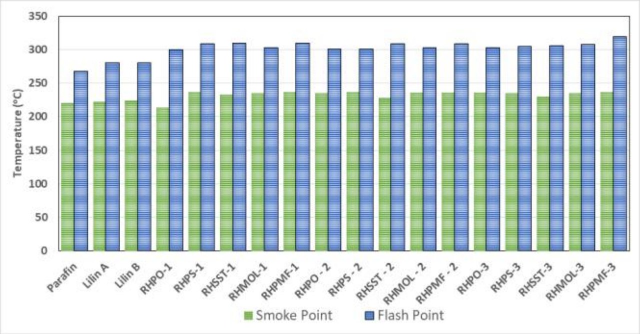


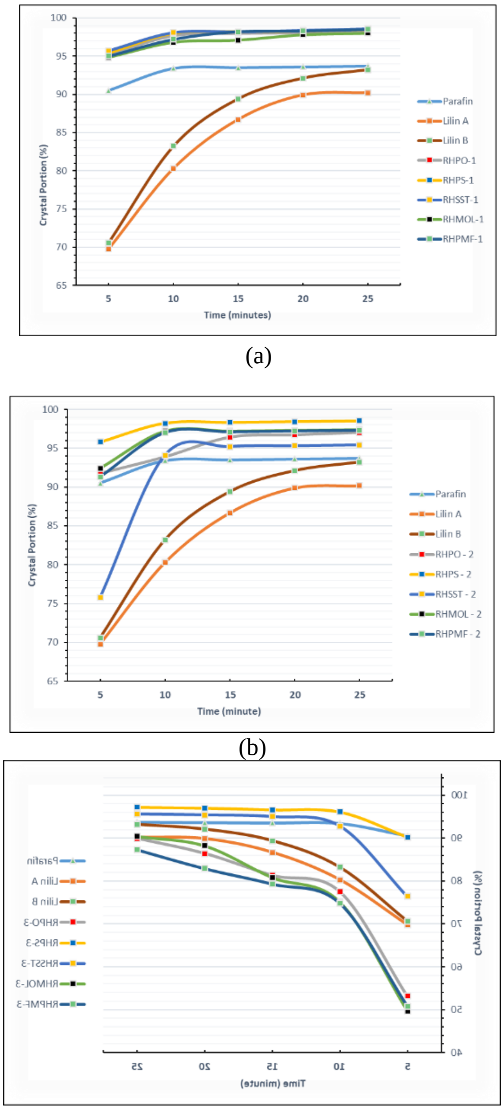
Figure 6. Smoke point and flash point of hydrogenated products

d. Crystallization Rate

Figure 7 shows the crystallization rates for all the hydrogenated products. It can be concluded that the crystallization rate was affected by the treatments of the hydrogenation process and thus will affect the crystallization rate of wax produced. Hydrogenated oils from treatment 1 were generally faster to crystallize, followed by treatment 2. Meanwhile, for treatment 3, although the crystallization rate was similar to wax and paraffin, it was slower than treatments 1 and 2.

e. Melting Rate

The melting rate indicates how fast the wax will melt. Consumers will certainly prefer wax that melts slowly or does not burn quickly compared to wax that melts quickly or burns quickly. Figure 8 shows that the RHPMF-3 was the hydrogenated oil with the highest melting rate, which has an average melting rate of 13.9%. Compared to hydrogenated products with treatment 1, which tend to be slower to melt, with a rate of 1.6 - 2% per minute. On the other hand, the commercial wax sample had a melting rate of 6.8 - 7.0% per minute, which was the same as paraffin.



(a)

(b)

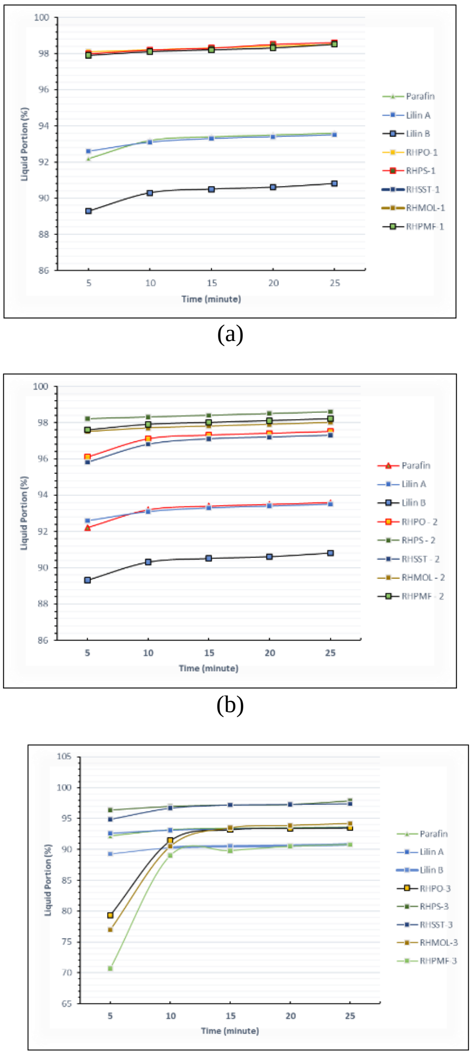
(c)

Figure 7. Crystallization rate: (a) treatment 1, (b) treatment 2, (c) treatment 3

3.2. *Candle Making and Characterization*

a. Combustion Diameter

Table 1 shows the wax diameter after burning. From Table 1, it is shown that wax with a high melting rate, such as RHPMF-3, will provide the highest melting diameter. Meanwhile, the hydrogenated products tended to be very slow, and the melting diameter was half that of paraffin. Wax in treatment 1 had a high crystallization rate and low melting rate compared to treatments 2 and 3, which caused the burn diameter to be smaller.



(a)

(b)

(c)

Figure 8. Melting rate: (a) treatment 1, (b) treatment 2, (c) treatment 3

Table 1. Wax diameter after burning

Candle product	Wax diameter after burning (mm)		
	5 min	10 min	15 min
Paraffin	10	20	25
Candle A	10	20	25
RHPO-1	5	7	14
RHPS-1	5	7	14
RHSST-1	5	7	14
RHMOL-1	5	7	14
RHPMF-1	5	7	14
RHPO-2	5	11	18
RHPS-2	5	11	18
RHSST-2	5	11	18
RHMOL-2	5	11	18
RHPMF-2	5	11	18
RHPO-3	5	9	15
RHPS-3	5	9	15
RHSST-3	5	11	28
RHMOL-3	5	11	28
RHPMF-3	5	11	28

b. Height of flame

Figure 9 shows that all waxes produced from hydrogenated oils have a flame height that is worse than commercial wax or paraffin, which means other alternatives must be done to increase the flame. High flame stability in a range of 30 mm to 40 mm indicates that the wax can provide good performance in terms of brightness, stability, and durability, which are important attributes for commercial wax in the market. Figure 9 also shows that there was no significant influence between the type of hydrogenation process and the type of ingredient preparation on the flame of the resulting wax.

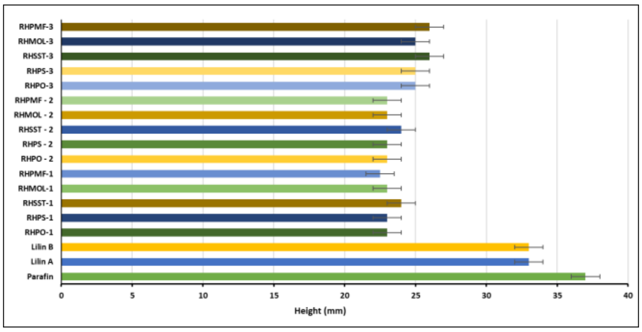


Figure 9. Height of the flame of wax

c. Burning Time

From Figure 10, several products with palm oil-based raw materials showed good performance compared to commercial wax and wax made from paraffin, with a burning time of 90 minutes. This wax was even 3 times better than commercial wax and 1.5 times better than paraffin wax.

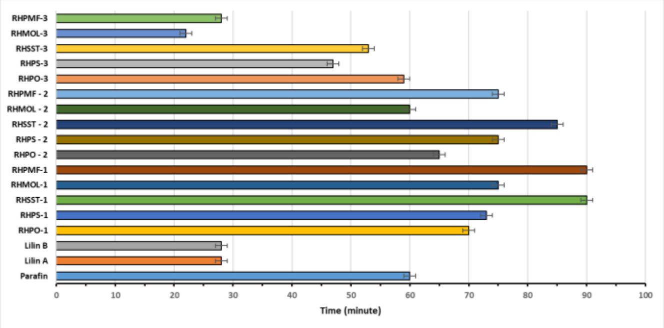


Figure 10. Burning time of various types of wax

d. Wax Texture and Hardness

Based on the testing results in Figure 11, RHPO-1 had the highest hardness and was even farther compared to commercial paraffin and wax. RHMOL-3 and RHPMF-3 tended to have the softest characteristics. Penetration testing of RHPMF-3 and RHMOL-3 showed that these waxes were easier to break, followed by RHSST-3. Even if broken by hand, this wax will break easily, become greasy, and become wet. The wax from paraffin was more resistant to pressure because it was more elastic, so when tested under pressure, this wax provided different elasticity.

Meanwhile, wax from the hydrogenation process of palm oil was the hardest, not having elasticity that makes it difficult to shape, and as a result, when dropped, this wax will be easier to break. Waxes with high stearic acid content commonly produce more solid wax.

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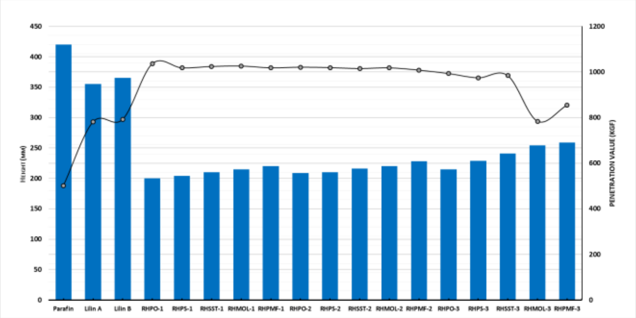

Figure 11. The results of wax hardness testing

4. CONCLUSION

The hydrogenation process of palm oil derivatives has a significant impact on the quality of hydrogenated products, which ultimately has a significant impact on the wax produced. The wax from a fully hydrogenated product physically looks stronger, more solid, and harder, having higher durability/stability, higher melting point and flash point, and low melting rate. However, wax produced from a hydrogenated product will have a shorter flame when burning/applied. Thus, it is less suitable for use in a wax application for lighting. Meanwhile, hydrogenated products from partial hydrogenation, either due to limited reaction time or limited amount of hydrogen used, have different characteristics, either in crystallization rate, melting rate, wax hardness, lighting produced, and so on. Based on the results of this study, it can be concluded that hydrogenated palm oil derivatives can be used as edible waxes to replace paraffin in wax applications. The selection is based on the availability of raw materials.

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