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Thermal and Physical Properties of Crude Palm Oil with Higher Oleic Content

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Abstract: Interspecific hybridization of oil palms (*E. guineensis* × *E. oleifera*) was initially exploited to provide disease resistance and, consequently, increased oleic acid content. Besides the growing importance of this cultivar to the market, there is little information about this oil's properties. In this context, this study aimed to determine a comprehensive physicochemical and thermal characterization of hybrid palm oil (HOPO) compared with the better-known African palm oil (APO). Differences in the distribution of fatty acids, carotenoids, and tocols were observed. Minor differences in density and viscosity were found between the oils, with no relevance for the materials' processing design. Nevertheless, HOPO showed unique crystallization behavior, which potentially can affect industrial operations, such as fractionation. HOPO did not present the two thermal characteristic regions of APO, attributed to olein and stearin fractions. The HOPO demonstrated a decrease in the melting point of more than 3 °C in relation to APO, and a reduction in the crystallization point of more than 6 °C. Furthermore, besides the higher content of unsaturated fatty acids, HOPO was more stable than APO due to a higher antioxidant content. These results could be useful to establish operation conditions for processes using palm oil from hybrid oil palm.

Keywords: crude palm oil; interspecific hybridization; melting; crystallization; carotenes

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1. Introduction

Palm oil has great importance in the world economic scenario, being the most used vegetable oil source. In fact, according to the United States Department of Agriculture, in 2020, palm oil world production was 75 million tons [1], surpassing the production of soybean, sunflower, and canola oil and accounting for one-third of vegetable oil production [2]. It has a wide application in industries, especially in food and pharmaceutical industries [3], also being one of the main alternatives for biofuel production in the following years [4].

Most commercial oil palms used are obtained from *E. guineensis*, an African species with high productivity per hectare, though very susceptible to fatal yellowing disease [5]. Alternatively, *E. oleifera*, an oil palm native from Central and South America, shows high resistance to this disease and other pests; however, it is not commercially exploited due to its low productivity. In this sense, the hybridization between both species (*E. guineensis* × *E. oleifera*) can generate an oil palm as productive as the African species and resistant to diseases like the American species [6].

Besides the disease resistance, hybridization also changes palm oil's chemical composition. Palm oil extracted from a hybrid cultivar showed an intermediary fatty acids profile compared to its two parental species [7]. Commonly, *E. guineensis* oil presents about 50% of saturated fatty acids, i.e., 44% of palmitic, 5% stearic, and traces of myristic, in addition to unsaturated fatty acids which correspond to approximately 40% oleic and 10% linoleic acids [8]. Regarding *E. guineensis* × *E. oleifera* oil, the fatty acids profile corresponds mainly to oleic (56%), palmitic (27%), linoleic (11%), and stearic (3.5%) acids [6]. The Codex Committee on Fats and Oils recommends commercializing such oil with the denomination of palm oil with higher oleic content [9].

In the literature, there are several studies regarding hybrid oil palm trees and their oils' characterization. The studies describe the cultivar's growth and its vegetative characteristics and characterization regarding oil composition in terms of fatty acids, acylglycerols, and minor compounds [5,7,8,10–12]. All those previously mentioned studies regard chemical composition and fatty acid profile. However, there is a lack of scientific studies on the processing and physical properties of palm oil from the hybrid cultivar.

Undeniably, the fatty acid profile determination assumes great importance since it is related to vegetable oils' physical properties. The wide range of food and non-food applications is due to palm oil's unique fatty acid composition, which allows its easy fractionation [13–15]. Under controlled thermal conditions, palm oil can be fractionated into two components in a single step: a solid fraction (stearin, which contains more trisaturated triacylglycerol and will crystallize) and a liquid fraction (olein, which contains less trisaturated triacylglycerol, remaining liquid) [2,13].

This fractionation process can determine some of the chemical and physical properties of the oleins and stearins produced. In the food industry, those two fractions have different applications; for instance, olein may be used as frying oil and stearin as a butter substitute [16]. Each fraction's yield highly depends on the triacylglycerol composition and palm oil's crystallization behavior [13,17,18]. Besides, fatty acids also play an important role in terms of their effects on human health. An oil with a high content of monounsaturated fatty acids, such as oleic acid, can lower bad cholesterol levels and protect against heart disease [5,6]. On the other hand, saturated fatty acids, such as palmitic acids, are related to a higher incidence of cardiovascular diseases and obesity [7,8]. African palm oil (APO) has a balanced composition between saturated and unsaturated fatty acids; hybrid palm oil (HOPO), in turn, has a high content of unsaturated fatty acids, especially oleic acid [9] Furthermore, palm oil is widely used in non-food industries, including its application other than food as hydraulic oil, drilling fluid, transmission fluid and lubricants, being fully biodegradable [19].

Several physical properties which infer process design also depend on the fatty acid composition. For instance, density and viscosity, essential fluid properties for the design of pipes, accessories, and equipment utilized in the oil industry, are highly dependent on the length and degree of fatty acids' unsaturation [20,21]. The composition of fatty acids attached to a triacylglycerol molecule determines its thermal behavior. The different molecular structures of triacylglycerols, with their different chemical characteristics, manifest their physical states at different temperatures, giving the oil crystallization and melting behavior [18]. The more saturated the fatty acids adhered to the triacylglycerol, the faster the crystallization processes. Besides the importance of palm oil for the industry and the growing interest in cultivating hybrid oil palms, there is a lack of physical and thermal properties for palm oils with higher oleic content.

In this context, this work aims to determine the physicochemical and thermal properties of commercial hybrid palm oil obtained from an interspecific hybrid cultivar (*E. oleifera* × *E. guineensis*). The results were compared to the better-known and widely commercially exploited African palm oil (*E. guineensis*).

2. Material and Methods

2.1. Physicochemical Characterization

Commercial crude oils obtained from two different oil palms—*Elaeis guineensis* and the interspecific hybridization of *Elaeis guineensis* × *Elaeis oleifera*, here so-called African

(APO) and hybrid palm oil (HOPO), respectively—were kindly supplied by Denpasa (Santa Bárbara do Pará, Brazil). The oil samples were characterized regarding standard quality parameters, following the AOCS official methods [22]: iodine value and unsaponifiable matter (Cd 1c-85 and Ca 6a-40). Total carotene content, expressed as β-carotene, was determined by measuring the absorbance at 446 nm of samples homogenized and diluted in hexane (Spectrophotometer Cary 5000, Varian, Palo Alto, CA, USA) [23]. All analyses were performed in triplicate. Acylglycerols were quantified by the Agilent 7890B gas chromatography system, following the AOCS official method (Santa Clara, CA, USA) (Cd 11b-91).

α- and β-carotene quantification: The chromatographic analysis was carried out in HPLC (Shimadzu, Kyoto, Japan) equipped with a quaternary pump (LC-20AD), a degasser, a UV-Vis DAD (SPD-M20A) detector, and an autoinjector (SIL-20A). The oven was set at 40 °C, and chromatograms were obtained at 450 nm. The compound separation was performed using a C30 column (Develosil, Hinode, Japan, 250 mm × 4.6 mm; 5 μm particle size). Elution gradient was a mixture of acetonitrile and methanol (0.05% triethylamine–TEA) and Tetrahydrofuran (THF) with a flow rate of 1.2 mL min^{-1}, in which the gradient only changed the concentrations of methanol and acetonitrile. The channels of the mobile phase were acetonitrile (A), methanol (0.05% TEA) (B), and tetrahydrofuran (C). Gradient proceeded as follows: (C) remained isocratic at 5% during all the analysis, and all the compositions varied linearly; from 0–10 min, (A) concentration varied from 95 to 65%, and (B) channel went from 0 to 30%; 10–20 min compositions varied to 55% (A) and 40% (B) and remained isocratic up until 60 min of the run; 60–65 min, the composition was set back to 65% (A) and 30% (B) and 65. After 65 min, (A) raised to 95% and (B) went down to 0%. Samples consisted of approximately 50 mg of crude oils diluted in 1 mL of acetone. The injection volume was 10 μL. The quantification was performed using Sigma-Aldrich α- and β-carotene standards (St. Louis, MO, USA).

Tocols quantification: Tocopherols and tocotrienols quantification was performed by UHPLC without sample pre-treatment, as previously reported [24]. The chromatographic analysis was performed using a Waters Acquity SQD/UPLC system (Milford, MA, USA), equipped with a PDA detector, a single-quadrupole mass spectrometer system with Electrospray Ionization interface, automatic injector, a quaternary pump, and a column oven. An Acquity UPLC BEH C18 column, 1.7 μm, 2.1 mm × 100 mm, was used (Milford, MA, USA). Instrument control and data acquisition were performed using the MassLynx software. Chromatographic separation was achieved by gradient elution using different proportions of Solvent A (methanol: water: ammonium hydroxide 99:1:0.1 *v*/*v*/*v*) and Solvent B (isopropanol). The flow rate was 0.2 mL min^{-1} up to 6 min, followed by 0.15 mL min^{-1} from 6 to 10 min, and 0.2 mL min^{-1} until 15 min. The elution gradient was as follows: 0–6 min 100% A; 6.01–8 min 100% B; 8.01–15 min 100% A. The injection volume was 5 μL, and the column temperature was 25 °C. Calibration curves were performed using commercial standards of α, β, γ, and δ tocopherols (Merck, Kenilworth, NJ, USA), and α, γ, and δ tocotrienols (Chromadex, Los Angeles, CA, USA).

Main fatty acids analysis: The oleic, linoleic, and linolenic determination were performed by ^{1}H nuclear magnetic resonance (NMR). A Bruker BioSpin GmbH spectrometer (Billerica, MA, USA) was used, operating in a magnetic field of approximately 14T with a 5 mm PABBO BB/19F-1H/D Z-GRD probe Z114607/0139, observing the cores from ^{1}H to 600.17 MHz. Samples were prepared using about 30–40 mg of each oil dissolved in 2 mL of deuterated chloroform containing tetramethylsilane (TMS), used as a reference. The acquisition parameters were: spectral width of 12,019.2 Hz, relaxation delay of 1 s, number of scans 16, acquisition time of 2.7263 s, and pulse width of 10.0400 Hz. The experiment was conducted at 25 °C. The ^{1}H NMR spectrum was analyzed in the MestRenova 12® software (Mestrelab Research, Billerica, MA, USA), using the TMS signal at 0.00 ppm. The integrals of the signs were used to calculate concentrations. The ^{1}H NMR technique employed in this work can quantify unsaturated fatty acids, the main influence in palm oil's physical properties, in a more straightforward and fast way than the traditional method by gas chromatography [21,22,25].

2.2. Physical and Thermal Properties

Density: Oil samples' densities were measured using a densimeter Rudolph Research Analytical DDM 2910 (New Jersey, NJ, USA) in the range of 20 to 80 °C at 10 °C intervals. The standard deviations of the measurements in the densimeter after proper calibration were 1×10^{-4} g cm^{-3}.

Viscosity: Viscosities of the oils were determined using 10 mL samples in the range of 20 to 80 °C at 10 °C intervals at atmospheric pressure using an automated rheometer Brookfield LV® (Middleboro, MA, USA) equipped with a spindle SC-34. The temperature uncertainty was 0.1 °C. Approximately 12 g of sample was under 100 rpm and 28 shear rate s^{-1} during 25 min. The viscosity data were obtained using Rheocalc® software (Version 2.4, Brookfield Engineering Laboratories, Middleboro, MA, USA) and the calculated standard deviations were ±1%.

Differential scanning calorimetry (DSC): The thermal behavior of oil samples was analyzed in duplicate using a differential scanning calorimeter coupled to an automated cooling TAC-60i (Shimadzu DSC-60, Kyoto, Japan) [26]. Oil samples of 3–4 mg were placed in aluminum pans sealed and analyzed under dynamic nitrogen atmosphere at 50 mL min^{-1}. Samples were equilibrated at 80 °C, cooled at 5 °C/min to −80 °C, and maintained at this temperature for 2 min to eliminate the thermal history; then, the samples were heated at 5 °C/min to 80 °C, and finally cooled at 5 °C/min to −80 °C. For oil thermal behavior, just the final heating and cooling data were considered.

Thermal gravimetric analysis (DTG): The thermogravimetric analyses of HOPO and APO were performed in DTG-60H equipment (Shimadzu, Kyoto, Japan). Samples of 3–4 mg were placed in platinum pans under a nitrogen flow of 50 mL min^{-1} and analyzed at a heating rate of 5 °C/min from 30 to 460 °C. Analyses were carried out using the TA software (Shimadzu, Kyoto, Japan) [20].

3. Results and Discussion

3.1. Chemical Characterization of the Oils

Table 1 presents the physicochemical characterization of HOPO and APO. Total carotene content was 830 and 524 mg/kg for HOPO and APO, respectively, practically within the range reported for African palm oil (500–1000 mg/kg) [27]. In APO, the β-carotene concentration was 380 mg/kg, while in HOPO it was 504 mg/kg. The α-carotene concentration was 51 mg/kg in APO and 60 mg/kg in HOPO. The concentration of α- + β-carotenes combined corresponded to 69 and 82% of the total carotenes for HOPO and APO, respectively. According to the literature, all quality parameters and carotene contents met the requirement expected for palm oil [12,28].

Regarding tocopherols, HOPO and APO presented 136.4 and 167.5 mg/kg of α-tocopherols, respectively. On the other hand, β-, γ- and δ-tocopherols were not detected in both oils, as usual for palm oil [12]. All classes of tocotrienols were present in HOPO and APO, i.e., 128.6 and 117.1 mg/kg of α-tocotrienol; 603.5 and 413.6 mg/kg of β-/γ-tocotrienols; and 15.2 and 29.8 mg/kg of δ-tocotrienol, respectively. The tocopherol/tocotrienol ratio of HOPO and APO was 18% and 29%, respectively. A similar ratio of about 20% was previously reported for an oil obtained from palm trees [29]. Moreover, Mozzon et al. [12] described similar results for HOPO, in which tocotrienols were found in high incidence, and the α-isomer was the most abundant tocopherol. Tocotrienols possess neuroprotective, anti-cancer and cholesterol-lowering properties that are often not ascribed to tocopherols [30].

Table 1. Physicochemical characteristics of hybrid (HOPO—*E. oleifera* × *E. guineensis*) and African (APO—*E. guineensis*) palm oil.

	HOPO	**APO**
ACYLGLYCEROLS (% W/W)		
MONOACYLGLYCEROL	0.21	0.24
DIACYLGLYCEROL	3.96	4.54
TRIACYLGLYCEROL	93.73	91.44
FATTY ACIDS PROFILE (%)		
OLEIC	56.34	44.81
LINOLEIC	7.35	7.65
LINOLENIC	2.06	1.28
FREE FATTY ACIDS (%, PALMITIC)	2.1 ± 0.09	3.8 ± 0.08
IODINE VALUE (G/100 G) *	67.7	52.3
UNSAPONIFIABLE MATTER (MG/G)	2.3 ± 0.2	1.3 ± 0.3
TOTAL CAROTENE (MG/KG)	830 ± 6	524 ± 4.4
α-CAROTENE	60	51
β-CAROTENE	504	380
TOCOLS (MG/KG)	883.3	728.0
α-TOCOPHEROL	136.4	167.5
β/γ-TOCOPHEROL	0	0
δ-TOCOPHEROL	0	0
α-TOCOTRIENOL	128.6	117.1
β/γ-TOCOTRIENOL	603.5	413.6
δ-TOCOTRIENOL	15.2	29.8

* calculated referring to fatty acid composition previously reported by Ribeiro, Almeida, Neto, Abdelnur, and Monteiro (2018).

The unsaturated fatty acids with the highest occurrence in both oils were oleic, linoleic, and linolenic (Table 1). Those correspond to 65.6 and 53.7% of total fatty acids, corroborated by previous reports [6]. Palmitic acid is mostly responsible for completing the fatty acids composition [5–7,12,28]. This fatty acid composition is vital to explain the differences in vegetable oils' physical properties described hereafter. Additionally, vegetable oil composition may vary slightly with cultivation conditions, raw material maturation, and storage time. Thus, this complete chemical characterization is important to guarantee the authenticity of studied oils and register the composition at the analysis moment.

3.2. Physicochemical Properties

The density of palm oils measured at temperatures from 20 to 80 °C to cover a is given in Table 2. The values follow the Codex Alimentarius Standard for palm oil (0.889–0.895 g mL^{-1} at 20 °C) [31]. The fatty acid composition can explain the slight difference between oils' density since hybrid palm oil has about 65% unsaturated fatty acids, while African has nearly 50% [6]. In fact, unsaturated fatty acids are denser than saturated ones [22]. Nevertheless, the differences in density found between hybrid and African oils are negligible for process design purposes. The viscosities of palm oil measured at temperatures from 40 to 80 °C are exhibited in Table 3. At temperatures lower than 40 °C, palm oils were at a semi-solid state, preventing viscosity measures. The viscosity of HOPO presented a smaller variation with temperature—a result of the higher content of unsaturated fatty acids [21].

3.3. Thermal Analysis of the Oils

The thermal profile of the oils, particularly the melting and crystallization events, is plotted in Figures 1 and 2. Due to the complex composition of triacylglycerols (TAGs) in the palm oil [28], several endothermic events, almost simultaneous, were observed in the heating program for both palm oils. In particular, two major endothermic events with a few sub-peaks can be identified (Figures 1 and 2 and Table 3). A similar thermal profile has been described for APO [28,32].

Table 2. Densities and viscosities of palm oils as a function of temperature *.

T (°C)	HOPO		APO	
	Density (g/cm^3)	Viscosity (cP)	Density (g/cm^3)	Viscosity (cP)
20	0.91369	-	0.91450	-
30	0.90637	-	0.90680	-
40	0.89866	40.19	0.89739	43.79
50	0.88663	27.59	0.88509	25.19
60	0.88396	20.4	0.88040	18.6
70	0.87849	15.6	0.86810	13.8
80	0.87174	12.0	0.86411	10.8

* Standard uncertainty: density < 0.0001 kg/m^3, viscosity ± 1%.

Table 3. Thermal events APO and HOPO obtained from the differential scanning calorimetry (DSC) curves.

		1° Peak	AD	2° Peak	AD	3° Peak	AD
APO	Tm	5.32	0.33	18.68	0.50	-	-
	TRm	−30.2 up to 13.5	-	13.42 up to 38.67	-	-	-
	Tc	−49.095	0.25	1.555	0.09	15.61	0.88
	TRc	−63.32 up to −39.55	-	−39.55 up to 5.88	-	5.88 up to 17.89	-
HOPO	Tm	2.47	0.33	11.36	0.83	-	-
	TRm	−30.0 up to 8.5	-	−0.05 up to 33.29	-	-	-
	Tc	−47.67	0.42	−4.81	0.25	9.98	0.84
	TRc	−71.88 up to 39.01	-	−39.01 up to 1.82	-	1.82 up to 12.29	-

T_m is the melting point of the most ascendant peak; T_{Rm} is the range temperature of melting transition; T_c is the crystallization temperature; T_{Rc} is the range temperature of crystallization transition; AD is the absolute deviation of the duplicates.

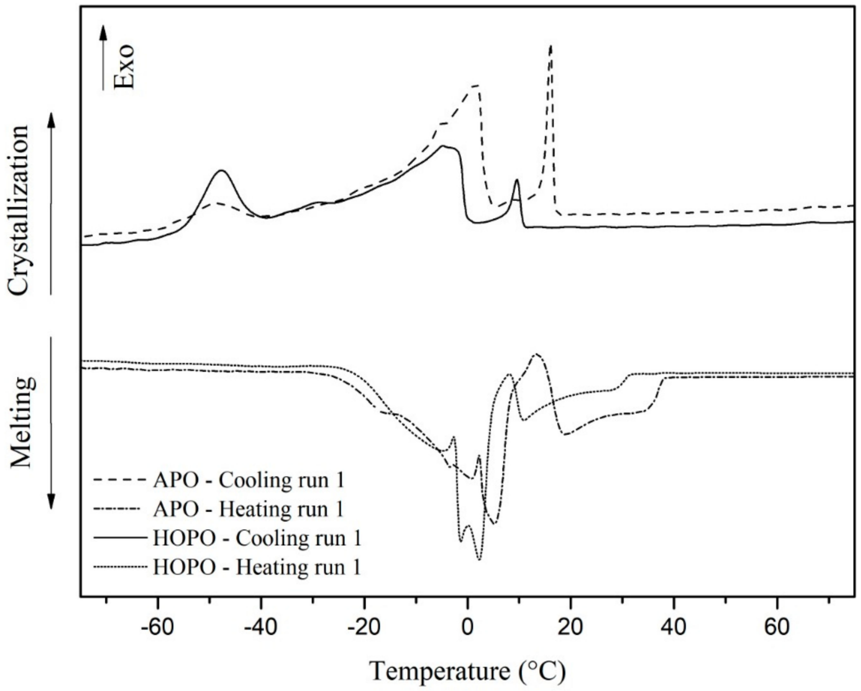

Figure 1. DSC curves in a heating and cooling program of APO and HOPO for run 1.

DSC data of heating showed the first event from −30.2 °C to 13.5 °C (enthalpy 39.27 kJ·kg^{-1}) for APO and from −30.0 to 8.5 °C (enthalpy 23.40 kJ·kg^{-1}) for HOPO, which are related to the contribution of the unsaturated triacylglycerols melting, such as UUU or SUU (ABC = fatty acid composition of TAG, U unsaturated fatty acid, S saturated fatty acid) [28,33]. This first broad endotherm event in both palm oils also indicates the presence of OOO, POL, and PLP compounds (P palmitic, O oleic and L linoleic acids). Moreover, the melting peak found in APO at 5.48 °C evidenced the presence of triolein compounds (melting temperature at 5.0 °C) [34]. The second endothermic event (Figure 1) is more pronounced in APO and should be related to the saturated triacylglycerols' contribution (SSS or SSU) [28].

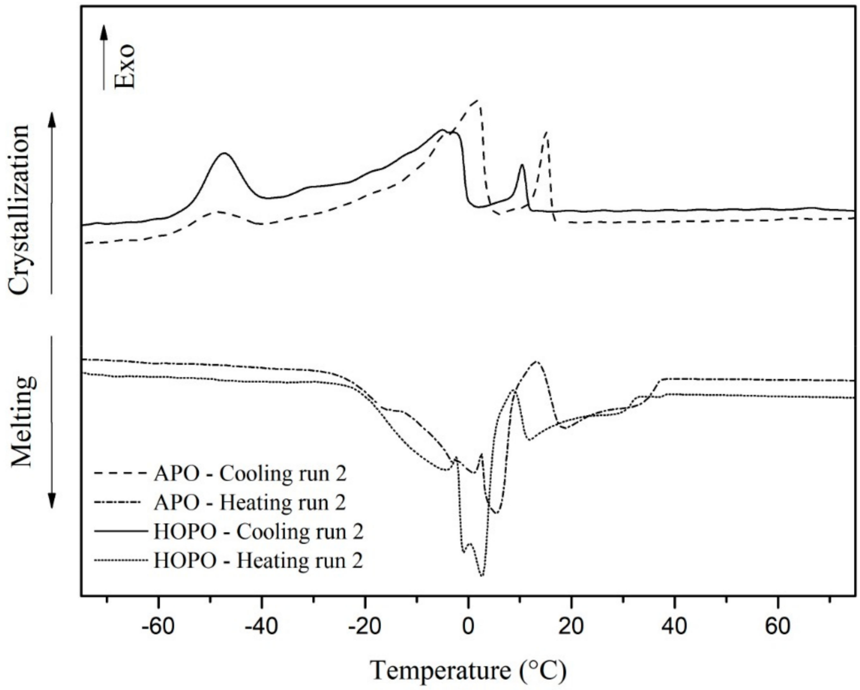

Figure 2. DSC curves in a heating and cooling program of APO and HOPO for run 2.

Table 3 shows the melting peaks and the range temperature of the endothermic events for both compounds. HOPO exhibited lower temperatures for the endothermic peaks than APO. This behavior could be related to the fact that the APO has about 50% of unsaturated fatty acids composition, while HOPO has around 65%. In fact, unsaturated compounds present lower melting points than saturated ones [28,32,34,35].

The DSC crystallization profiles for APO and HOPO obtained by the samples' cooling program are presented in Figures 1 and 2 and Table 3. APO showed three crystallization peaks (Tc), which are −49.09 °C, 1.55 °C and 15.61 °C. A similar crystallization profile was noted for HOPO (Table 3). Additionally, in the case of APO, a third peak involving less energy, ranging from 5.88 to 17.99 °C, can be attributed to the presence of TAGs such as SSS and SSU triacylglycerols [28]. The exotherm observed at lower temperatures, which in the case of APO ranges from 5.88 to 63.32 °C, and in the case of HOPO involves temperatures from 1.82 to 71.88 °C, is possibly related to unsaturated fatty acids (for example, UUU and SUU). These findings explain the broadest and most intense exotherm observed in HOPO, which showed a higher content of unsaturated fatty acids [33]. Similar behavior was previously reported for crude palm oil, involving two sharp peaks of crystallization obtained at temperatures slightly higher than those found here [8,33].

The mass loss related to the decomposition of palm oils occurred in two steps (Figure 3). The thermal decomposition profile of both oils is similar, including a slow first step of mass loss from 180 to 300 °C, and a fast second step of mass loss from 180 to 440 °C, which accounts for more than 90% of the sample mass loss. Nevertheless, a difference in the kinetic of the first step of mass loss is noted among the oils (Figure 3). The first temperature range is related to the degradation of polyunsaturated fatty acids, followed by monosaturated and saturated fatty acids [36]. Even with a higher concentration of unsaturated fatty acid, HOPO is slightly more stable than APO, showing a slower first decomposition step. This thermal profile is probably due to the high amounts of antioxidant compounds, such as carotenes and tocols. The same stability improvement was reported for several commercial edible oils, such as olive, soybean, and canola [34].

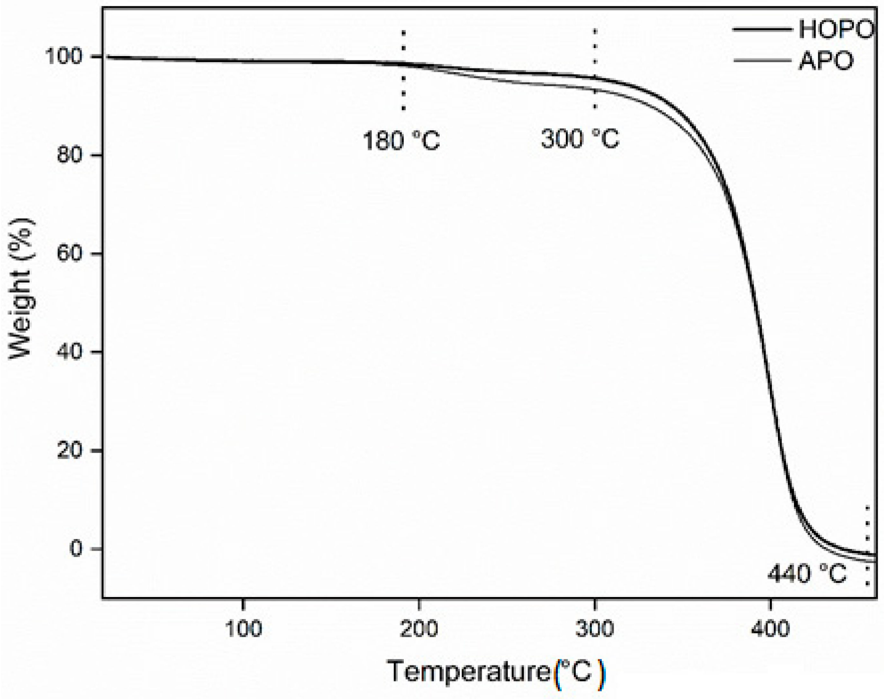

Figure 3. TGA curves of palm oil APO and HOPO.

4. Conclusions

This study determined the physicochemical, physical, and thermal properties of hybrid palm oil obtained from an interspecific hybrid cultivar (*E. oleifera* × *E. guineensis*), which was compared to the palm oil obtained from the African cultivar (*E. guineensis*). The oils presented chemical differences; however, both showed an appropriate chemical profile required for commercial exploitation. Moreover, small differences in density and viscosity were found between the oils, with no relevance for the materials' processing design. Nevertheless, the thermal analysis demonstrated that the hybrid species' oil presented distinguished melting/crystallization behavior, affecting industrial operations, such as fractionation and olein and stearin yields. Furthermore, besides the higher content of unsaturated fatty acids, HOPO was more stable than APO due to a higher antioxidant content. The results presented here are useful to establish operation conditions for processes using palm oil from hybrid oil palm, a cultivar that is arousing the industry's interest due to its resistance to pests.

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