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Effects of different storage conditions on the oxidative stability of crude and refined palm oil, olein and stearin (*Elaeis guineensis*)

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Abstract

Crude palm oil (CPO), refined palm oil (RPO), refined palm olein (RPOL) and refined palm stearin (RPS) were stored in three conditions: kept away in dark (at 20-25 °C, acclimatized environment); in a refrigerator (4-8 °C); and at room temperature (26-32 °C), exposed to natural light. Free fatty acids (FFA; %), peroxide value (meq O_2/kg), induction period (h), total carotenoids (ppm) and color measurements (CIELab) were analyzed to determine stability of oils every months until 12 months. All of the crude/refined initial oils were of good quality, except for one sample of CPO. Storage at 26-32 °C and exposure to light intensified the oxidative reactions. The estimated shelf life of CPO, RPO, RPOL and RPS, when stored at 20-25 °C and in the dark, would be approximately 6, 9, 9 and 12 months, respectively. The best quality oils was found stored at 4-8 °C when compared to those stored in other storage conditions.

Keywords: crude palm oil; olein; stearin; storage.

Practical Application: The study has an influence for practice of the storage, a storage time and conditions, in addition to discuss to most important criteria to get quality products. Palm oil is widely used due to its high stability and considering the cultural and public health importance of consuming.

1 Introduction

Palm oil (*Elaeis guineensis*) has outclassed soybean oil during the last decade to become the most produced vegetable oil in the world, accounting for 57% of vegetable oil exports in the markets (Oil World, 2013). It is a versatile plant with cosmopolitan economic importance in Nigeria, Malaysia, Brazil and several West African countries (Akusu et al., 2000; Frank et al., 2011; Almeida et al., 2013). In Brazil, the first palm oil agro-industrial projects were installed in the northeastern region of the State of Pará in the 1960s, and its cultivation there has undergone a major expansion in the last decade (Villela et al., 2014).

Palm oil has a balanced fatty acid composition in which the level of saturated fatty acids is almost equal to that of the unsaturated fatty acids. Palmitic acid (44-45%) and oleic acid (39-40%) are the major component acids along with linoleic acid (10-11%) and a trace amount of linolenic acid (Mba et al., 2015). This composition allows palm oil to be fractionated into two major fractions: a liquid oil (65-70%), palm olein (m.p. 18-20 °C), and a solid fraction (30-35%), stearin (m.p. 48-50 °C). In addition to the real fat fraction, crude palm oil contains minor components (1%) such as sterols (250-620 ppm), squalene (200-600 ppm) (Gunstone & Lin, 2011), and carotenoids (500-700 ppm), pigments which are responsible for the reddish orange color (Gee, 2007). Furthermore, it is the richest source of tocotrienols among all vegetable oils (Sambanthamurthi et al., 2000; Edem, 2002).

The quality and stability of palm oil are the main factors influencing its acceptability and market value, as well an minimize the degration process during the deep friyng (Almeida et. al., 2017). One of the most important indicators of the keeping quality of oil is its oxidative stability (Tan et al., 2017). In its turn, the oxidative stability of vegetable oils depends on temperature, light, oxygen, metals, enzymes, the presence of antioxidants or proxidants, fatty acid composition, and the use of oxygen permeable packages (Pristouri et al., 2010; Ahmad et al., 2011). In addition, Frank et al. (2011), reported that deteriorative changes in palm oil during storage are caused by the type of storage material, light, air and autocatalytic hydrolysis by lipolytic microorganisms and water content.

Different chemical mechanisms are responsible for the oxidation of edible oils during storage: autoxidation and photosensitized oxidation. Autoxidation is a reaction between unsaturated fatty acids, regardless of whether they are in their free state or esterified as a triglyceride molecule and oxygen. These reactions originate hydroperoxides, which are rapidly decomposed to aldehydes, ketones, alcohols, hydrocarbons, esters, furans and lactones (Choe & Min, 2006; Adetola et al., 2016). Changes in oil quality during storage conditions inappropriate are still a major issue from the health perspective.

In Bahia-Brazil, retailers and wholesale dealers store palm oil for sale in open air, elevated temperature, packed in opaque plastic and exposed to natural or artificial light. The aims of this study was to evaluate the effects of different storage conditions and **storage times** on palm oil and the quality characteristics of its fractions.

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2 Materials and methods

2.1 Obtaining samples

Crude palm oil (CPO), refined palm oil (RPO), refined palm olein (RPOL) and refined palm stearin (RPS) were donated from Agropalma Company (Belém-Pará-Brazil); these samples were all produced during the same period. All were sent by mail to Federal University of Bahia (Salvador-Brazil) in cardboard boxes packed in opaque plastic (250 mL) of polyethylene high-density (PEHD). According to the information provided by this producer, the shelf life for CPO, RPO, RPOL and RPS is 12, 9, 9 and 12 months, respectively. The samples contained Tert Butyl Hydroquinone/TBHQ (INS319) in the proportion of 180 ppm.

2.2 Experimental design

A factorial experiment was designed: storage time x storage condition. The levels applied were four storage times (sampling period of 3 months: 0, 3, 6, 9 and 12 months) and three storage conditions, a closed area kept away in dark (20-25 °C, acclimatized environment), a refrigerator (4-8 °C), and at room temperature exposed to natural light (26-32 °C), measuring carefully using thermometer. Every analysis, a sample of each oil was withdrawn from its original packaging. Three distinct lots of each oil, in triplicate, were studied: crude palm oil (CPO), refined palm oil (RPO), refined palm olein (RPOL) and refined palm stearin (RPS). This was done to reduce error from specific lot condition and not due to the representative general oil process condition.

The study room temperature ranged between 26 and 32 °C, with an average of 29.5 °C, and relative humidity between 74.37 and 89.83%, checked the it every time. These storage conditions are similar to those observed at the points of sale of oils.

2.3 Analytical determinations

Free fatty acids (FFA %) and peroxide value (PV meq O_2/kg)

Fatty acids and peroxide were analyzed in triplicate according to AOCS Ca 5a-40 and AOCS Cd 8-53 (American Oil Chemists' Society, 2003), respectively.

Induction period (h)

The oxidative stability of the oil samples was determined with a Rancimat 743 (Metrohm AG, Switzerland). In brief, 3 g of the vegetable oil were weighed into the reaction vessel and heated at 120 °C with an air flow of 20 L h^{-1}. The volatile products released during the oxidation process were collected in a flask containing distilled water. The oxidation process was recorded automatically by measuring the change in conductivity of the distilled water due to the formation of volatile compounds and the oil stability index (OSI), which is expressed in hours (h) (Läubli & Bruttel, 1986). At each time point, eight oil samples were analyzed simultaneously by the equipment. Each sample was analyzed in duplicate.

Total carotenoids (ppm)

Crude palm oil samples (±0.2-0.3 g) were dissolved in petroleum ether and quantified in a Lambda 25 UV-Vis spectrophotometer (Perkin Elmer, Singapore) at 450 nm with an absorption coefficient (A1%1 cm) of 2592 (Rodriguez-Amaya & Kimura, 2004). The analysis was performed in triplicate.

Color measurement (CIELab)

The color was measured using quartz cells 2 mm thick in a Chroma Meter CR-400 (Konica Minolta Sensing Inc., Osaka, Japan) with D65 illuminant and an angle of view of 2°. The results were expressed in terms of lightness (L*), red–green characteristics (a*), blue–yellow characteristics (b*), hue angle (h_{ab}) and chroma (C*); $h_{ab} = \tan{-1}\ (b^*/a^*)$ and $C^* = [(a^{*2} + b^{*2})^{1/2}]$. Next, color difference (ΔE) was calculated as $\Delta E = \{(Lo-L)^2+(ao-a)^2+(bo-b)^2\}^{1/2}$), where Lo, ao and bo are the color parameters of the fresh oil samples (Time 0) and 12 meses. Each color value reported was the mean of three determinations at 22-24 °C (Andreu-Sevilla et al., 2008).

2.4 Statistical analysis

Statistical analyses were conducted with SPSS (Statistical Package for the Social Sciences) 13.0.1 for Windows. As Levene's test for equalizing variances was statistically significant for all of the parameters, Tamhane's methods were utilized ($p \leq 0.05$). Regression analysis was performed to evaluate the relationships between the time and degradation parameters. The model fitting was judged by a coefficient of determination (R^2), which represents the proportion of variability that has been accounted for the polynomial regression equation. The linear correlation between two parameters was assessed by Spearman´s correlation coefficient (r).

3 Results and discussion

3.1 Free Fatty Acids (FFA)

Free fatty acid (FFA %) content is the most widely used criterion for determining the quality of palm oil (Almeida et al. 2013). Codex 210 (Codex Alimentarius, 2013) and Brazilian legislation (Brasil, 2005) established a maximum concentration value of FFA up to **5.0**% for CPO and up to **0.3**% for RPO in oleic acid. Thus, according to these norms, all of the fresh oils (zero months of storage) were within the set limits (Tables 1-4). The results revealed that the production of FFA in the fresh CPO were lower (**1.2-1.6**%) compared with the CPO made in Pará (**2.2-2.5**%) (Almeida et al., 2013). This finding may indicate that the palm fruits were handled gently and processed rapidly after harvest and sterilized with stream in order to limit lipase activity (Vincent et al., 2014).

The results revealed that FFA (%) increased in all the CPO at different stored conditions and during all storage times. Tagoe et al. (2012) demonstrated that processed fresh fruit oils had initial acidity of **0.5**% and **3**% after 12 months storage. The increase in content of FFA should be caused by endemic species of microorganisms that are introduced into the oil during various stages of processing and transport within the plant (Nkpa et al., 1990). Furthermore, this result was found for all treatment conditions (Table 1); this

indicates fluctuations over time, similar to the results observed by Frank et al. (2011). This does not necessarily express a real decrease, as unsaturated FFA may undergo subsequent chemical reactions such as peroxidation and generate secondary products that cannot be detected while assaying acidity.

A time limit was estimated at which each CPO surpassed the maximum value for FFA (%) defined by CODEX 210 (Codex Alimentarius, 2013) and Brazilian legislation (Brasil, 2005). The maximum estimated time periods were 14, 15 and 18 months for CPO storage between 26-32 °C, 20-25 °C and 4-8 °C, respectively.

The results of all determinations conducted for refined palm oil (RPO), refined palm olein (RPOL) and refined palm stearin (RPS) are presented in Tables 2-4, respectively. After 12 months of storage at 26-32 °C under natural light, all refined oils exceeded the upper limits of FFA (0.3%) established by Codex 210 (Codex Alimentarius, 2013), with a determination coefficient (R^2) of 0.99. These results indicate a stronger relationship between the storage time and the evolution of acid value. On the contrary, for refined oils stored at 20-25 °C and 4-8 °C, the FFA (%) did not exceed the adopted limit of 0.3% after 12 months of storage (Codex Alimentarius, 2013; Brasil, 2005).

The results for the FFA % of the refined oils for all storage conditions and times (Tables 2, 3 and 4) are much higher when compared to soy and canola oils packed in polyethylene terephthalate (PET), stored in the dark, with an average temperature of 19 °C for 375 days (0.04-0.15% and 0.06-0.08%, respectively) (Ahmad et al., 2011). Indeed, palm oils tend to have higher moisture content and increased microbial load as storage time increases, allowing the hydrolytic reactions responsible for the formation of free fatty acids (Akusu et al., 2000; Tagoe et al., 2012).

3.2 Peroxide Value (PV)

Another important parameter used to assess the quality of palm oil was the peroxide value (PV), which is an indicator of the level of lipid oxidation. Must not exceed the upper limit (15 meq O_2/kg) established by Codex 210 (Codex Alimentarius, 2013). CPO (storage at 4-8 °C) showed an initial value of 8.7 meq O_2/kg, approximately 15 times higher than the initial values for the samples stored at 26-32 °C and 20-25 °C (Table 1). These results may indicate inadequate storage because the FFA % these oils did not differ from the others ($p \geq 0.05$) (Table 1), demonstrating efficient refining. According to the process, even though the oils are manufactured on the same day, the samples are stored in different drums, which could influence these results.

According to Farhoosh et al. (2009), for recently refined oils, the peroxide value should be too close or equal to zero and should not surpass 0.5 meq O_2/kg. The results showed that some fresh refined oils had exceeded that limit (Tables 2, 3 and 4). This can be attributed to the time gap between production and analysis, which was approximately 10 days.

The evolution of PV indicates that the exposure to light and elevated temperature potentiate the formation of lipid peroxide molecules (Nkpa et al., 1990). It was observed that RPO and RPS exceeded the peroxide value (15 meq O_2/kg) defined by Codex 210 (Codex Alimentarius, 2013) after the 3-month storage period, while CPO and RPOL surpassed the limit after 6 months of storage (Table 1 and 3). Warner & Nelsen (1996), suggested

Table 1. Evolution of the FFA (%), peroxide value (meq O_2/kg), induction period (h), total carotenoids (ppm) and color (CIELab) of crude palm oil (CPO) during the storage time and in different conditions.

Months of storage	Free fatty acids (% oleic acid)	Peroxide value (meq O_2/Kg)	Induction period (h)	Total carotenoids (ppm)	L*	a*	b*	C*	h_{ab}	ΔE (*)
26-32 °C										
0	1.17^{a} (0.03)	0.61^{a} (0.05)	17.04^{a} (0.60)	766.83^{a} (27.33)	30.79^{a} (0.18)	14.28^{a} (0.04)	20.57^{a} (0.16)	25.04^{a} (0.11)	55.22^{a} (0.29)	
3	3.06^{bf} (0.07)	9.90^{b} (0.18)	13.59^{ab} (0.13)	715.36^{a} (18.12)	30.34^{ad} (0.11)	14.91^{b} (0.07)	19.95^{a} (0.38)	24.90^{ab} (0.34)	53.21^{b} (0.42)	0.99
6	2.50^{cg} (0.07)	21.54^{c} (0.27)	8.69^{ac} (0.08)	600.13^{b} (25.35)	31.24^{ae} (0.05)	13.77^{c} (0.06)	21.19^{a} (0.20)	25.27^{a} (0.19)	56.99^{c} (0.16)	0.92
9	2.32^{dg} (0.07)	29.39^{d} (0.40)	5.45^{ad} (0.14)	440.02^{c} (11.70)	32.39^{bf} (0.03)	12.31^{d} (0.07)	23.73^{b} (0.04)	26.73^{bc} (0.07)	62.58^{d} (0.12)	4.05
12	3.14^{ef} (0.00)	33.79^{e} (0.50)	0.02^{ae} (0.00)	252.42^{d} (1.53)	34.00^{c} (0.02)	09.12b^{e} (0.07)	25.93^{c} (0.23)	27.49^{c} (0.25)	70.62^{e} (0.08)	8.10
20-25 °C										
0	1.78^{a} (0.08)	0.58^{a} (0.04)	15.01^{a} (0.12)	757.41^{a} (17.59)	30.57^{a} (0.10)	14.55^{a} (0.17)	20.05^{a} (0.36)	24.77^{a} (0.39)	54.03^{a} (0.18)	
3	2.94^{bf} (0.06)	2.55^{b} (0.02)	16.04^{a} (0.12)	720.34^{a} (18.37)	30.49^{a} (0.09)	14.40^{a} (0.10)	19.81^{ab} (0.17)	24.49^{ab} (0.19)	54.00^{a} (0.05)	0.29
6	2.89^{cf} (0.02)	6.15^{c} (0.12)	14.88^{ab} (0.59)	722.08^{ac} (4.94)	30.69^{ab} (0.23)	13.86^{ab} (0.30)	19.30^{ac} (0.88)	23.76^{a} (0.88)	54.29^{a} (0.74)	1.03
9	2.47^{df} (0.13)	16.76^{df} (0.07)	14.59^{ac} (0.01)	692.41^{ad} (4.92)	31.88^{b} (0.08)	12.23^{b} (0.10)	22.29^{bc} (0.47)	25.42^{a} (0.46)	61.25^{b} (0.33)	3.48
12	2.85^{ef} (0.01)	16.49^{ef} (0.09)	11.65^{bc} (0.19)	563.47^{b} (4.52)	33.03^{c} (0.02)	10.39^{c} (0.00)	24.36^{c} (0.00)	26.54^{ac} (0.26)	66.80^{c} (0.12)	6.48
4-8 °C										
0	1.58^{a} (0.05)	8.68^{a} (0.03)	5.18^{a} (0.16)	648.27^{a} (1.61)	31.05^{a} (0.07)	13.62^{a} (0.11)	21.13^{a} (0.32)	25.14^{a} (0.33)	57.19^{a} (0.19)	
3	2.67^{bf} (0.01)	12.12^{b} (0.15)	5.46^{a} (0.16)	605.82^{bd} (0.00)	31.12^{a} (0.06)	13.46^{a} (0.10)	21.76^{a} (0.33)	25.59^{a} (0.33)	58.26^{b} (0.20)	0.65
6	2.87^{cd} (0.02)	16.12^{cf} (0.13)	5.92^{a} (0.01)	593.84^{cd} (0.00)	31.74^{b} (0.05)	13.00^{b} (0.07)	20.87^{ab} (0.12)	24.58^{ab} (0.13)	58.08^{ab} (0.10)	0.96
9	2.58^{df} (0.08)	16.97^{d} (0.08)	5.32^{a} (0.21)	574.33^{de} (8.05)	31.71^{abc} (0.15)	12.10^{abc} (0.35)	21.55^{a} (1.23)	24.72^{a} (1.24)	60.67^{abc}(0.70)	1.71
12	2.57^{ef} (0.05)	16.15^{ef} (0.02)	5.01^{a} (0.00)	553.89^{e} (0.00)	32.41^{c} (0.08)	11.60^{c} (0.05)	23.49^{ac} (0.04)	26.20^{ac} (0.05)	63.72^{c} (0.08)	3.39

Data are presented as mean ± (standard deviation). The same letter in each column represents no significant difference between sample values by the Tamhane test ($p \leq 0.05$). L* (lightness); a* (negative values indicate green and positive values indicate red, -a/+a); b* (negative values indicate blue and positive values indicate yellow, +b/-b); C* (choma) and h_{ab} (hue angle). Data ΔE (color difference) for each storage condition (temperature range) were obtained by comparing the data of each month (3, 6, 9 and 12) with month 0 (control).

a classification for the oxidation level of vegetable oils based on the evolution of the peroxide value. According to these authors, oils with a peroxide value between 3 and 5 meq O_2/kg can be considered to be of low oxidation; between 10 and 12 meq O_2/kg are of moderate oxidation; and between 16 and 18 meq O_2/kg are of a high level of oxidation. All of the samples stored between

Table 2. Evolution of the FFA (%), peroxide value (meq O_2/kg), induction period (h) and color (CIELab) of refined palm oil (RPO) during the storage time and in different conditions.

Months of storage	Free fatty acids (% oleic acid)	Peroxide value (meq O_2/Kg)	Induction period (h)	L*	a*	b*	C*	h_{ab}	ΔE (*)
26-32 °C									
0	0.11^{a} (0.00)	00.18^{a} (0.00)	15.79^{a} (1.12)	39.93^{a} (0.67)	-1.47^{a} (0.06)	4.98^{a} (0.12)	5.19^{a} (0.10)	106.48^{a} (1.00)	
3	0.15^{a} (0.00)	10.50^{b} (0.05)	10.75^{ab} (0.33)	39.06^{a} (0.22)	-0.82^{a} (0.04)	2.76^{ab} (0.04)	2.88^{ab} (0.05)	106.55^{a} (0.62)	2.47
6	0.16^{ab} (0.01)	19.69^{abc} (0.63)	06.85^{ac} (0.37)	40.50^{a} (0.03)	-0.87^{a} (0.04)	2.89^{a} (0.09)	3.02^{a} (0.88)	106.68^{a} (0.74)	2.25
9	0.23^{a} (0.00)	31.63^{abc} (0.82)	02.39^{ad} (0.16)	40.72^{a} (0.11)	-0.61^{a} (0.03)	2.36^{ac} (0.02)	2.44^{ac} (0.02)	104.37^{a} (0.62)	2.87
12	0.40^{ac} (0.01)	58.36^{c} (0.12)	00.03^{ae} (0.00)	40.92^{a} (0.11)	-0.39^{a} (0.02)	1.66^{a} (0.01)	1.70^{a} (0.02)	103.19^{a} (0.67)	3.63
20-25 °C									
0	0.14^{a} (0.01)	0.29^{a} (0.00)	14.14^{a} (0.08)	40.26^{a} (0.11)	-1.62^{a} (0.02)	5.45^{a} (0.01)	5.69^{a} (0.02)	106.60^{a} (0.24)	
3	0.17^{a} (0.01)	1.00^{ab} (0.00)	14.22^{a} (0.33)	40.45^{b} (0.02)	-1.68^{ab} (0.04)	5.34^{ad} (0.05)	5.60^{ad} (0.06)	107.43^{a} (0.25)	0.23
6	0.21^{a} (0.01)	3.13^{a} (0.08)	14.44^{a} (0.67)	40.37^{ab} (0.07)	-1.55^{ac} (0.04)	5.40^{ae} (0.02)	5.62^{ae} (0.02)	106.01^{a} (0.34)	0.14
9	0.13^{a} (0.01)	7.70^{b} (0.06)	11.56^{a} (0.18)	40.15^{ab} (0.31)	-1.43^{ad} (0.04)	5.67^{be} (0.06)	5.85^{be} (0.05)	104.20^{a} (0.52)	0.31
12	0.22^{a} (0.01)	13.82^{c} (0.05)	10.12^{a} (0.08)	40.75^{ab} (0.06)	-0.43^{ae} (1.54)	4.89^{ce} (0.02)	5.07^{ce} (0.03)	105.26^{a} (0.10)	1.40
4-8 °C									
0	0.13^{a} (0.01)	0.44^{a} (0.01)	14.99^{a} (1.09)	40.29^{a} (0.06)	-1.66^{a} (0.02)	5.39^{a} (0.02)	5.63^{a} (0.03)	107.10^{a} (0.10)	
3	0.16^{a} (0.01)	0.52^{a} (0.04)	15.27^{abd} (0.05)	40.49^{a} (0.23)	-1.54^{be} (0.01)	5.19^{ab} (0.05)	5.41^{a} (0.05)	106.54^{a} (0.23)	0.31
6	0.16^{a} (0.01)	0.69^{a} (0.02)	13.74^{a} (0.28)	40.47^{a} (0.11)	-1.60^{ce} (0.01)	5.35^{a} (0.04)	5.58^{ab} (0.04)	106.66^{a} (0.19)	0.19
9	0.13^{a} (0.01)	0.63^{a} (0.02)	10.50^{ad} (0.18)	40.86^{a} (0.02)	-1.71^{ae} (0.03)	5.57^{a} (0.04)	5.83^{a} (0.04)	107.03^{a} (0.22)	0.60
12	0.23^{a} (0.01)	1.14^{a} (0.00)	10.62^{ac} (0.03)	40.84^{a} (0.04)	-1.39^{de} (0.02)	4.80^{ac} (0.02)	5.00^{ac} (0.01)	106.18^{a} (0.26)	0.85

Data are presented as means (standard deviation). The same letter in each column represents no significant difference between sample values by the Tamhane test ($p \leq 0.05$). L* (lightness); a* (negative values indicate green and positive values indicate red, -a/+a); b* (negative values indicate blue and positive values indicate yellow, +b/-b); C* (choma) and h_{ab} (hue angle). Data ΔE (color difference) for each storage condition (temperature range) were obtained by comparing the data of each month (3, 6, 9 and 12) with month 0 (control).

Table 3. Evolution of the FFA (%), peroxide value (meq O_2/kg), induction period (h) and color (CIELab) of refined palm olein (RPOL) during the storage time and in different conditions.

Months of storage	Free fatty acids (% oleic acid)	Peroxide value (meq O_2/Kg)	Induction period (h)	L*	a*	b*	C*	h_{ab}	ΔE (*)
26-32 °C									
0	0.04^{a} (0.00)	00.52^{a} (0.04)	12.88^{a} (0.10)	40.67^{a} (0.09)	-1.36^{a} (0.04)	4.77^{a} (0.03)	4.96^{a} (0.04)	105.95^{a} (0.37)	
3	0.17^{a} (0.00)	00.45^{a} (0.02)	09.19^{bc} (0.04)	40.66^{a} (0.06)	-1.07^{a} (0.03)	3.45^{abcd} (0.09)	3.61^{ab} (0.08)	107.23^{ab} (0.79)	1.35
6	0.18^{a} (0.01)	25.44^{a} (0.47)	04.10^{cde} (0.21)	40.73^{a} (0.07)	-0.74^{ab} (0.02)	2.79^{bcd} (0.03)	2.88^{b} (0.03)	104.80^{ab} (0.25)	2.08
9	0.23^{a} (0.01)	44.91^{ab} (3.27)	00.92^{de} (0.01)	41.13^{a} (0.04)	-0.53^{a} (0.02)	1.93^{c} (0.01)	2.01^{abc} (0.01)	105.34^{ab} (0.55)	2.99
12	0.33^{a} (0.03)	85.29^{b} (1.31)	00.03^{e} (0.00)	41.46^{a} (0.21)	-0.25^{ac} (0.01)	1.55^{d} (0.18)	1.57^{abd} (0.18)	099.24^{b} (1.01)	3.50
20-25 °C									
0	0.07^{a} (0.00)	00.59^{a} (0.02)	14.74^{a} (0.03)	40.59^{a} (0.11)	-1.70^{a} (0.03)	5.55^{a} (0.07)	5.80^{a} (0.07)	107.01^{a} (0.07)	
3	0.15^{a} (0.00)	00.43^{ab} (0.00)	13.90^{ab} (0.33)	40.59^{ab} (0.08)	-1.72^{abc} (0.02)	5.61^{abc} (0.06)	5.87^{abc} (0.06)	106.99^{a} (0.18)	0.06
6	0.16^{a} (0.01)	05.69^{b} (0.01)	12.14^{a} (0.30)	40.28^{a} (0.14)	-1.41^{bc} (0.02)	5.15^{abc} (0.10)	5.34^{abc} (0.10)	105.35^{a} (0.01)	0.58
9	0.16^{a} (0.00)	08.92^{abc} (0.20)	10.72^{a} (0.56)	40.85^{ab} (0.09)	-1.41^{abc} (0.04)	4.98^{bc} (0.02)	5.18^{bc} (0.02)	105.83^{a} (0.36)	0.69
12	0.17^{a} (0.01)	14.64^{c} (0.04)	09.34^{ac} (0.30)	42.30^{b} (0.01)	-1.40^{c} (0.01)	4.98^{c} (0.01)	5.17^{c} (0.00)	105.72^{a} (0.12)	1.83
4-8 °C									
0	0.29^{a} (0.00)	00.43^{a} (0.04)	14.80^{a} (0.28)	40.78^{a} (0.02)	-1.66^{a} (0.02)	5.40^{a} (0.01)	5.65^{a} (0.01)	107.10^{a} (0.15)	
3	0.17^{a} (0.00)	00.59^{a} (0.00)	13.92^{a} (0.41)	40.79^{abc} (0.07)	-1.66^{a} (0.03)	5.50^{a} (0.15)	5.75^{a} (0.15)	106.77^{a} (0.11)	0.10
6	0.40^{a} (0.01)	00.86^{a} (0.03)	14.97^{ab} (0.16)	40.25^{b} (0.06)	-1.54^{a} (0.03)	5.23^{a} (0.05)	5.46^{a} (0.04)	106.43^{a} (0.37)	0.57
9	0.24^{a} (0.00)	00.95^{a} (0.00)	10.49^{a} (0.33)	40.81^{abc} (0.05)	-1.69^{a} (0.03)	5.47^{a} (0.02)	5.72^{a} (0.02)	107.17^{a} (0.17)	0.08
12	0.23^{a} (0.01)	01.69^{a} (0.01)	09.97^{ac} (0.14)	42.06^{c} (0.01)	-1.52^{a} (0.02)	5.23^{a} (0.00)	5.45^{a} (0.00)	106.26^{a} (0.15)	1.30

Data are presented as means (standard deviation). The same letter in each column represents no significant difference between sample values by the Tamhane test ($p \leq 0.05$). L* (lightness); a* (negative values indicate green and positive values indicate red, -a/+a); b* (negative values indicate blue and positive values indicate yellow, +b/-b); C* (choma) and h_{ab} (hue angle); Data ΔE (color difference) for each storage condition (temperature range) were obtained by comparing the data of each month (3, 6, 9 and 12) with month 0 (control).

Table 4. Evolution of the FFA (%), peroxide value (meq O_2/kg), induction period (h) and color (CIELab) of refined palm stearin (RPS) during the storage time and in different conditions.

Months of storage	Free fatty acids (% oleic acid)	Peroxide value (meq O_2/kg)	Induction period (h)	L*	a*	b*	C*	h_{ab}	ΔE (*)
26-32ºC									
0	0.09^{a} (0.00)	0.72^{a} (0.00)	17.62^{a} (0.12)	40.51^{a} (0.02)	-0.78^{a} (0.02)	2.87^{a} (0.01)	2.98^{a} (0.02)	105.21^{a} (0.24)	
3	0.09^{a} (0.00)	11.36^{ad} (0.47)	8.78^{abcd} (0.42)	40.64^{ac} (0.20)	-0.48^{a} (0.05)	1.69^{a} (0.09)	1.76^{a} (0.10)	105.62^{a} (0.96)	1.22
6	0.19^{a} (0.00)	22.43^{bde} (0.13)	2.93^{bcd} (0.04)	40.88^{ae} (0.10)	-0.37^{a} (0.02)	1.65^{a} (0.05)	1.69^{a} (0.04)	102.70^{a} (0.68)	1.34
9	0.45^{a} (0.00)	41.40^{adf} (0.00)	0.03^{cd} (0.00)	41.47^{bce} (0.06)	-0.47^{ab} (0.01)	1.37^{a} (0.07)	1.45^{a} (0.07)	108.81^{a} (0.83)	1.81
12	1.48^{a} (0.00)	85.38^{c} (0.02)	0.02^{d} (0.00)	40.22^{ad} (0.05)	-0.23^{ac} (0.01)	1.14^{b} (0.01)	1.16^{a} (0.01)	101.42^{a} (0.49)	1.84
20-25 °C									
0	0.09^{a} (0.00)	00.88^{a} (0.00)	17.17^{a} (0.04)	40.76^{a} (0.04)	-0.69^{a} (0.01)	2.59^{a} (0.08)	2.68^{a} (0.08)	104.89^{a} (0.30)	
3	0.09^{a} (0.00)	02.04^{a} (0.00)	15.97^{a} (0.66)	40.86^{a} (0.00)	-0.71^{ac} (0.00)	2.65^{abd} (0.00)	2.78^{ab} (0.00)	104.75^{a} (0.00)	0.10
6	0.09^{a} (0.00)	03.40^{ab} (0.07)	13.87^{a} (0.21)	40.96^{a} (0.07)	-0.73^{ad} (0.02)	2.62^{a} (0.28)	2.88^{a} (0.02)	104.61^{a} (0.31)	0.21
9	0.30^{a} (0.00)	06.09^{ab} (0.14)	12.29^{a} (0.32)	41.61^{ab} (0.03)	-0.89^{bcde} (0.01)	2.99^{ac} (0.04)	3.12^{ac} (0.04)	106.49^{a} (0.30)	0.96
12	0.29^{a} (0.01)	05.01^{b} (0.02)	10.67^{b} (0.00)	40.89^{ac} (0.05)	-0.65^{aae} (0.02)	2.54^{ad} (0.02)	2.62^{ad} (0.02)	104.44^{a} (0.40)	0.14
4-8 °C									
0	0.09^{a} (0.00)	0.53^{a} (0.01)	17.28^{a} (0.51)	40.72^{a} (0.12)	-0.77^{a} (0.02)	3.06^{a} (0.08)	3.15^{a} (0.08)	104.20^{a} (0.15)	
3	0.11^{a} (0.00)	2.04^{a} (0.00)	15.92^{a} (0.20)	40.87^{ab} (0.07)	-0.90^{a} (0.02)	3.12^{a} (0.12)	3.25^{a} (0.12)	106.10^{a} (0.80)	0.17
6	0.13^{a} (0.00)	1.10^{a} (0.00)	16.36^{a} (0.13)	40.83^{ac} (0.04)	-0.82^{a} (0.01)	3.22^{ab} (0.01)	3.32^{ab} (0.01)	104.35^{a} (0.11)	0.20
9	0.27^{a} (0.00)	1.22^{a} (0.00)	16.01^{a} (0.27)	41.22^{ad} (0.21)	-1.00^{a} (0.03)	3.43^{a} (0.10)	3.57^{ad} (0.09)	106.28^{a} (0.58)	0.66
12	0.22^{a} (0.01)	1.07^{a} (0.01)	15.31^{b} (0.25)	41.20^{bcd} (0.10)	-0.67^{a} (0.01)	2.61^{ac} (0.02)	2.69^{ac} (0.02)	104.31^{a} (0.26)	0.67

Data are presented as means (standard deviation). The same letter in each column represents no significant difference between sample values by the Tamhane test ($p \leq 0.05$). L* (lightness); a* (negative values indicate green and positive values indicate red, -a/+a); b* (negative values indicate blue and positive values indicate yellow, +b/-b); C* (choma) and h_{ab} (hue angle). Data ΔE (color difference) for each storage condition (temperature range) were obtained by comparing the data of each month (3, 6, 9 and 12) with month 0 (control).

26-32 °C under natural light presented a peroxide value above 26 meq O_2/kg after the 6-month storage period and therefore may be considered to be highly oxidized.

After 9 months of storage at room temperature, the PV of CPO was lower than the other samples, indicating that the crude oil was more resistant to oxidation (Tables 1-4). This most probably can be attributed to the carotene content. Carotenoids could inhibit further decomposition of hydroperoxide by reacting with alkoxyl radicals RO, which are mainly responsible for the generation of volatile compounds. This can be inferred by the high negative linear correlation between carotenoids and PV ($r = - 0.963$; $p \leq 0.00$).

Furthermore, the PV results for CPO, RPO and RPOL storage at 20-25 °C were outside of specification (Codex Alimentarius, 2013) after 9, 12 and 12 month storage periods, respectively.

According to the results obtained, CPO stored at 4-8 °C, even those that began with a high PV, had increased by only 1.8 times by the end of a year of storage, with time having a strong influence ($R^2 = 0.98$). The results showed that for all refined samples of oil stored in that condition (Tables 2, 3 and 4), the PV were lower than the limit value establish in Codex 210 (Codex Alimentarius, 2013) and the Brazilian legislation (Brasil, 2005) after the 12 month storage period. Moreover, PV of the stearin samples stored at 20-25 °C and 4-8 °C did not exceed the adopted limits (10 meq O_2/kg) during the entire period of storage and had the lowest PV compared to other oils studied. Such differences may be due to the predominance of saturated fatty acids in its composition; these are less susceptible to oxidation (Edem, 2002; Choe & Min, 2006; Gunstone & Lin, 2011).

3.3 Changes in oxidative stability

The oxidative stability of oils was measured through the induction period and determined using the Rancimat method showed in Tables 1-4. As for the peroxide values, CPO samples stored at 4-8 °C also differ in this regard from the others (Table 1). They showed the lowest values reflecting the degree of oxidation (Table 1). The mean values for the initial induction period for RPO, RPLO and RPS were **15.0, 14.1** h and **17.3** h, respectively. None show a significant difference between the IT initial lots of RPO (Table 2) and stearin (Table 4), unlike olein (Table 3), whose lots were stored at 26-32 °C and 20-25 °C and differed significantly ($p \leq 0.04$). It is well known that this fraction has a higher tendency to oxidation due to the greater presence of unsaturated fatty acids (Zambiazi & Zambiazi, 2000).

It is clear that the oxidative deterioration of all of the oils proceeded significantly faster when stored in the elevated temperature and with exposure to light than under the other storage conditions (Choe & Min, 2006).

The induction period (IT) showed high value for the coefficient of determination ($R^2 = 0.99$) during storage for the refined oils kept under 20-25 °C, indicating a strong relationship between time and a reduction of IT in the samples. In this case, the IT remained high for all oils (from **9.3 to 11.3** h), confirming that these saw the highest oxidation resistance in a lower-temperature environment and in the dark (Borsato et al., 2012). The same occurred with the oil stored in the refrigerator (**10.0 to 15.3**-h), except for the CPO, which as previously mentioned, has begun to change (**5.2** h); however, that still remained stable over time (**5.0** hours) (Table 1), which has in their composition the carotenoids.

3.4 Total carotenoids and color

The Codex 210 (Codex Alimentarius, 2013) establishes a value for total carotenoids of 500-2000 ppm and thus, according this norm, all of the initial CPO analyzed were within range (**648-767** ppm). Studies of CPO produced in Pará-Brazil detected values between **939-940** ppm (Almeida et al., 2013).

In the selected lots of CPO, there were no significant differences in the amount of carotenoids between the initial oils subjected to three treatments ($p \geq 0.05$). However, in the initial oils stored at 4-8 °C, the carotenoid contents were smaller and inverse correlations were found between peroxide and β-carotene content ($r= -0.813$; $p = 0.00$) ($p \leq 0.05$). It was understood that the oxidation of carotenes is accelerated by hydro peroxides generated from lipid oxidation, leading to discoloration and bleaching; α- and β ionones, β-13 and β-14-apocarotenals and β-13-apocrotenone are among the carotenoids (Sambanthamurthi et al., 2000).

All CPO samples were located within the first quadrant, showing that a* and b* are positive. No significant changes in C and b* between the initial CPOs were observed. It was seen that their hue values (h_{ab}) were located in the zone of reddish-orange color (Table 1) (Tan et al., 2010). The color of these oils was distinctively more intense than that of the oils produced in Bahia, possibly because of better fruit processing conditions (Almeida et al., 2013). The variations in the mean values of h_{ab} among the initial CPO studied were significant ($p < 0.05$), and the h_{ab} value is very high for the oil storage at 4-8 °C when compared to the other storage conditions ($p \leq 0.05$), (Table 1), indicating a loss of orange color.

Tables 1-4 show the difference (ΔE) between the oils for different storage times and conditions. The parameter ΔE was chosen as an indicator of color fading because it allowed the concomitant changes of all color parameters to be taken into account. The visual threshold allowing an average observer to note the color difference is at least 3 CIELab units (Ceballos et al., 2003). According to this, the change in color of CPO samples stored at 26-32 °C and 20-25 °C became noticeable ($\Delta E \geq 3$) after 9 months of storage (Table 1). The ΔE of the CPO sample stored under refrigeration changed in 12 months. These results reflects the increase of L*, b* and h_{ab} and the decrease of a* (Table 1), which indicate that oxidation led to a loss of orange color in the oils. The value of lightness L* increased during the entire period of storage, as all oils became more transparent as an effect of degradation (Sikorska et al., 2007). Such color changes during storage were mainly caused by rapid photodegradation of the carotenoids groups, and the presence of oxygen in the headspace is a crucial factor in the β-carotene degradation (Rodriguez-Amaya & Kimura, 2004).

The color characteristics of the refined palm oils are shown in Tables 2-4. There is a lack in the literature concerning α, β -carotene pathways during refining practices and its importance in the quality of fully refined oils. The amount of carotenoids in the crude oil does not determine the residual color of the refined oil. Some studies suggest that in the finished oil, the color is mostly due to high molecular weight (HMW) compounds derived from oxidation reactions (Silva et al., 2014).

All refined oil samples were located within the second quadrant, showing a* negative and b* positive, which indicates the intensity of the greenish yellow color (Tables 2, 3 and 4).

It can be concluded that the color change phenomenon grew more intense at higher storage temperatures, with greater light exposure and with greater unsaturated fatty content (Sikorska et al., 2007; Pristouri et al., 2010).

4 Conclusion

The palm oils analyzed in this study presented good initial quality, in accordance with current legislation on free fatty acids, peroxide value and carotenoids, except for one CPO sample, which is attributed to inadequate storage.

Storage at room temperature and exposure to light intensified the oxidative reactions. These were characterized by a change in the color, an increase in free fatty acids and peroxide values, and a reduction in the induction period and the total carotenoids. Therefore, it is not recommended that any type of palm oil be stored in that condition.

It was seen that after 12 months, oils stored at 4-8 °C presented better oxidative stability when compared to the other storage conditions. Thus, in accordance with this study, it is recommended that palm oils be stored under refrigeration.

It should be noted that the crude palm oils in Bahia are marketed over at least 18 months and exposed to light and high temperatures and packaged in plastic transparent bottles without the addition of antioxidants. Therefore, in addition to these study findings, the results also demonstrate the need for specific legislation regarding palm oil and its components in conjunction with extensive and efficient inspection of their storage when sold.

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