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Processing practices and quality of crude palm oil produced on a small scale in Valença, Bahia, Brazil

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ABSTRACT: This study aimed to compare the quality of crude palm oils (CPO) produced from different processing methods in Brazil: traditional (T-CPOE), little-mechanized (LM-CPOE) and semi-mechanized extraction (SM-CPOE). A total of 57 CPO samples were studied. The peroxide value (PV), total carotenoids (TC), oxidative stability index (OSI_{120}), deterioration of bleachability index (DOBI), color (CIELab), total polar compounds (TPC) and its fractions, consisting of triacylglycerol polymers (TGP), dimers (TGD), oxidized monomers (oxTGM), diacylglycerols (DAG) and free fatty acids (FFA) were analyzed. Regardless of the extraction method, the PV, TC and OSI_{120} were found to be 3.1-7.6 $meqO_2$/kg, 375-598 ppm, and 0.6-3.3 h, respectively. T-CPOE samples demonstrated superior quality based on DOBI and TPC. The TPC in the T-CPOE samples were significantly lower (8.7-9.6%) than in the other samples (11.9-18.5%). In all methods, the levels of compounds in the TPC fractions were DAG ≈ FFA > oxTGM > (TGP + TGD). Fruit selection and T-CPOE equipment played crucial roles in reducing oil degradation.

KEY-WORDS*: Crude palm oil; Elaeis guineensis; Free fatty acids; Physicochemical quality; Total polar compounds.*

RESUMEN: ***Prácticas de procesamiento y calidad del aceite de palma crudo producido a pequeña escala en Valencia, Bahía, Brasil***. El objetivo de este estudio fue comparar la calidad de aceites de palma crudo (CPOs) producidos por diferentes métodos de procesamiento en Brasil: extracción tradicional (T-CPOE), poco mecanizada (LM-CPOE) y semimecanizada (SM-CPOE). Se estudiaron un total de 57 muestras de CPO. Se analizaron el índice de peróxidos (PV), contenido de carotenoides (TC), índice de estabilidad oxidativa (OSI_{120}), índice de deterioro de la capacidad de decoloración (DOBI), color (CIELab), compuestos polares totales (TPC) y sus fracciones, que incluyen polímeros de triacilgliceroles (TGP), dímeros (TGD), monómeros oxidados (oxTGM), diacilgliceroles (DG) y ácidos grasos libres (FFA). Independientemente del método de extracción, los resultados obtenidos para el PV, TC y OSI fueron 3,1-7,6 $meqO_2$/kg, 375-598 ppm, y 0,6-3,3 h, respectivamente. Según el DOBI y el TPC, las muestras T-CPOE presentaron mayor calidad. Los valores de TPC fueron más bajos (8,7-9,6%) que para el resto de las muestras (11,9-18,5%). En todos los métodos, los niveles de las fracciones TPC fueron DAG ≈ FFA > oxTGM > (TGP + TGD). La selección de frutos y el equipo T-CPOE fueron factores determinantes en la reducción de la degradación del aceite.

PALABRAS CLAVES*: Aceite de palma crudo; Ácidos grasos libres; Calidad isicoquímica; Compuestos polares totales; Elaeis guineensis.*

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1. INTRODUCTION

Palm oil is currently the major source of edible oil in the world (Gonzalez-Diaz *et al*., 2021), and is derived from the mesocarp of the oil palm fruit (*Elaeis guineensis Jacq.*). It exists in both crude (CPO) and refined (PO) forms. CPO contains approximately 39-44% palmitic acid, 39-40% oleic acid, 10-11% linoleic acid, and the rest is composed by 5% stearic acid and small proportions of lauric, myristic, palmitoleic, linolenic and arachidic acids (De Almeida *et al*., 2013; De Almeida *et al*., 2018; Feitosa *et al*., 2019). In tropical climates, PO is semi-solid and can be fractionated into two fractions: olein, which has a high content of unsaturated fatty acids, and stearin, which is rich in saturated fatty acids.

CPO also contains relatively high concentrations of tocopherols and tocotrienols (vitamin E), and carotenoids (500-700 ppm), 90% of which are present as α- and β-carotenes. In addition, β-carotene imparts the characteristic orange-red color of CPO (Gonzalez-Diaz *et al.*, 2021). Tocopherols, tocotrienols and carotenoids are all bioactive compounds that play an essential role in the durability of CPO because they have antioxidant properties (De Almeida *et al.*, 2013; de Almeida *et al.*, 2018; Feitosa *et al.*, 2019).

The production of crude palm oil (CPO) in Brazil is concentrated in the states of Pará and Bahia (De Almeida *et al.*, 2013; de Almeida *et al.*, 2018; de Almeida *et al.*, 2019; Feitosa *et al.*, 2019). Being the second state in CPO production, Bahia's oil palm groves are dominated by emergent palms of the *dura* variety (thin mesocarp, thick endocarp), which yields less oil than the *tenera* cultivar (thick mesocarp, thin endocarp) (De Almeida *et al.*, 2013). Within Bahia, the municipality of Valença stands prominently as one of the leading CPO producers. In addition, Bahia is also one of the main CPO consumption areas. CPO is used as a major cooking oil and a common ingredient in several traditional recipes such as *akara, moin-moin, vatapá, caruru and moquecas* (Feitosa *et al.*, 2019; Cardoso *et al.*, 2022).

Conventional and emerging technologies for CPO extraction have been reviewed recently (Chiew *et al.*, 2022). However, very little has been documented on the production of CPO in rural areas, where this is carried out by artisanal procedures without or with little mechanization. In this regard, most of the CPO produced in Bahia for human consumption is processed by smallholders who basically use traditional crude palm oil extraction (T-CPOE), little-mechanized crude palm oil extraction (LM-CPOE) or semi-mechanized crude palm oil extraction (SM-CPOE). Despite the scientific literature presenting simple methods of CPO extraction, the extraction methods used in Valença are quite rudimentary and they are not described in the literature. The different conditions applied in these extraction procedures may have different impacts on the CPO quality.

The aims of the present study were to report the processing practices carried out in the production of CPO by smallholders in Valença-Bahia and assess their impact on oil quality. The peroxide value, deterioration of bleachability index, oxidative stability index, total carotenoids, color parameters, the total content of polar compounds and their fractions differing in molecular weight were analyzed. This study can help policymakers encourage producers to move towards better structures based on the effects of processing on the quality of CPO.

2. MATERIALS AND METHODS

2.1. Data collection

The study was conducted at 'smallholders' processors in the municipality of Valença, a Brazilian city located on the coast of the state of Bahia, northeast region of Brazil.

An introductory meeting was held with the Association of Palm Oil Producers in Valença, the government representatives and the manufacturers of CPO extraction machinery. The latter were surveyed. The mill owners were selected according to their interest in participating in this study. Data were collected through verbal interviews with the producers and manufactures of CPO. The processes were observed and the workers were also interviewed during CPO extraction. Observations and interviews were conducted to gather pertinent data about the company's real conditions, products and existing technologies, equipment operation, degree and type of mechanization, safety and environmental issues associated with CPO extraction. All participants provided informed consent, which had previously been approved by the Ethics Committee of the School of Nutrition of the Federal University of Bahia (Protocol 2.027.540).

2.2. Sample collection

Nineteen smallholder oil palm fruit processing mills were visited (4 T-CPOE, 8 LM-CPOE and 7 SM-CPOE) in Valença during the palm fruit season. Specifically, oil sampling was performed from December 2017 to March 2018. Three visits were conducted to each mill, in intervals of 30 days, during which CPO samples were collected. The number of samples studied were 57 (12 T-CPOE, 24 LM-CPOE and 21 SM-CPOE). On the day of each visit, the researchers followed the entire extraction process and 1 L of bottled CPO was randomly selected the following day from each type of extraction according to the local procedure. This procedure aimed to collect cold samples. The oils were wrapped in aluminum foil to protect them from light. After collection, the samples were taken to the laboratory in thermal containers. Then, the samples were transferred to amber bottles, flushed with nitrogen and sealed. The bottles were stored at -20 °C prior to analysis.

2.3. Analytical methods

2.3.1. Peroxide value (PV)

The PV was determined in triplicate according to AOCS Cb 8b-90 method (AOCS, 2003).

2.3.2. Total carotenoids

The total carotenoids content was analyzed in triplicate by spectrophotometry (Perkin Elmer, Singapore) at 450 nm applying an absorption coefficient ($A^{1\%}_{1cm}$) of 2592. Two grams of freeze-dried oil sample were extracted with 50 mL cold acetone. The results were expressed in ppm (Cardoso *et al.*, 2022).

2.3.3. Color parameters

Color parameters were measured in triplicate using quartz cells of 2 mm thick in Chroma Meter CR-400 (Konica Minolta Sensing Inc., Japan) and expressed in terms of lightness (L*), red-green characteristics (a*), blue-yellow characteristics (b*), Hue angle (h°) and chroma (C*); $h° = \tan^{-1}(b^*/a^*)$ and $C^* = [(a^*2 + b^*2)^{1/2}]$. Each color value reported was the mean of three determinations at 22-24 °C (De Almeida *et al.*, 2018).

2.3.4. Deterioration of bleachability index (DOBI)

The DOBI was determined in triplicate by using the PORIM test (Kheang *et al.*, 2006). A known mass of the oil was dissolved in n-hexane (95%) and the absorbance was measured at 446 nm and 269 nm, using a spectrophotometer (Perkin Elmer, Singapore). The results were expressed as the ratio between their absorbances (446 nm / 269 nm). The DOBI rating was: excellent quality (DOBI >3.24), good quality (2.93-3.24), fair quality (2.31-2.92) and poor quality (1.68-2.30) (Gee, 1999).

2.3.5. Oxidative stability index (OSI) determined by the Rancimat test

The OSI of the oil samples was performed in triplicate using a Rancimat device, 743 model (Metrohm CH-9101, Herisau, Switzerland). Three grams of oil were weighed into the reaction vessel and the measurements were taken at 120 °C with an airflow of 10 L/h (De Almeida *et al.*, 2013; de Almeida *et al.*, 2018).

2.3.6. Analysis of total polar compounds (TPC)

The TPC was determined in duplicate by adsorption chromatography according to the IUPAC standard method (Dobarganes *et al.*, 2000) with slight modifications (Feitosa *et al.*, 2019).

2.3.7. HPSEC analysis of the polar compounds

The polar fractions obtained in the standard method were in turn analyzed in duplicate by high-performance size-exclusion chromatography (HPSEC) with refractive index detection to determine triacylglycerol polymers (TGP), triacylglycerol dimers (TGD), oxidized triacylglycerol monomers (oxTGM), diacylglycerols (DAG) and free fatty acids (FFA), i.e. groups of compounds with different molecular weight, according to (Dobarganes *et al.*, 2000).

2.4. Statistical analysis

Data were analyzed using IBM SPSS software package version 29.0 (Armonk, NY: IBM Corp). One-way ANOVA was applied. Multiple comparisons were made using Duncan's test or the non-parametric Gomes-Howell's test when the Levene's test for equality of variances was not fulfilled. *Spearman's* correlation was performed to assess the relationship between the different physicochemical parameters. Significance was considered at the 5% level.

3. RESULTS AND DISCUSSION

3.1. Processing practices in Valença-Bahia for CPO extraction

A flow chart diagram illustrating the operation units involved in each CPO extraction process is presented in Figure 1. Generally, the CPO extraction encompasses several stages, including splitting (shredding) and/or chopping of fresh fruit bunches (FFB), storage of cut bunches for fruit loosening, threshing to separate the fruit from bunches, sterilization, crushing, oil separation and clarification. These producers use vehicles or animals to transport the fruit bunches to the processing unit. All three methods share the common practice of processing fruits within a time frame that typically varies from three to six days after fruit harvesting. This relatively long time facilitates the separation of fruit from bunches, but favors and intensifies the oil acidity (De Almeida *et al.*, 2013; Nchanji *et al.*, 2013). In the T-CPOE method, threshing is carried out manually. In LM-CPOE, an axe or machete is employed for threshing, while in SM-CPOE, a mechanical thresher (4m x 0.8m x 1.6m) is utilized. This mechanical thresher employs either rotation or vibration to effectively separate the fruit from the bunches (Figure 1). The T-CPOE is normally performed by individuals who select the ripe fruits, remove the damaged ones, and manually wash them.

The sterilization process can be carried out using boiling water or steam, and it serves a dual purpose. Firstly, it softens the fruit, making oil extraction easier. Secondly, it deactivates lipolytic enzymes that can cause oil hydrolysis and oxidation, ultimately leading to a significant reduction in oil quality (Nchanji *et al.*, 2013). In the T-CPOE, the fruit (about 20 kg per batch) is cooked or sterilized in boiling water for 2 h using an aluminum pan (20 L). An open fire fueled by empty fruit bunches and fiber is employed for this purpose. In the LM-CPOE, a higher amount of fruit is processed per batch (about 200 kg) and the sterilization is also conducted in boiling water, but the duration ranges from 3 to 6 h, and iron drums (200 L) are utilized.

After this procedure, the water is poured onto the ground, and the fruits are cooled to room temperature. In the SM-CPOE, the FFB (2 tons) are sterilized with steam for 2.5-3 h using an iron tank (6 m x 3 m x 1.5 m) equipped with a discharge port. Then, an employee removes the cooked bunches from the tank using an iron hook and takes them to the mechanical thresher, which is driven by a 7.5-hp electric motor capable of processing 6 tons of fruit per hour, effectively separating the fruit from the bunches at high speed (Figure 1). In this method, the empty bunches slide down the front of the thresher and onto the floor, where they are subsequently removed by an employee using a wheelbarrow.

The palm fruit separated from the bunches proceeds to the digestion process, which involves both crushing and maceration to extract the oil (Nwakodo *et al.*, 2020). The LM-CPOE and SM-CPOE methods employ mechanized digesters. In the LM-CPOE process, a horizontal iron crusher measuring 2.4 m in length, equipped with 40 small iron blades (10cm x 5cm), is utilized. This crusher is powered by a 5-hp engine and has a processing capacity of 4 tons of fruit per hour. In the SM-CPOE, the digester is a 3-meter-long vertical metal tube containing 7 metal blades (30 cm x 7.5 cm). This digester is driven by a 5-hp engine coupled with a speed reducer. It can process up to 6 tons of fruit per hour. In the T-CPOE method, this is performed either by pounding the cooked fruit in large wooden mortars with wooden pestles or by foot trampling on the cooked, albeit cold, fruit (Figure 1).

The digested mash is transferred to a 20 L aluminum pan in the T-CPOE or to a cement tank with a capacity of 2 to 3 thousand liters in the LM-CPOE process. In both cases, oil separation is achieved through the addition of water. The volume of water added is twice the volume of the digested mash. Then, the mixture containing oil, seeds, fibers, and water is vigorously shaken by hand or using long wooden spoons. This agitation process continues until the oil separates from the mixture due to differences in density, rising to the surface, while the remaining components are decanted (Figure 1). In the T-CPOE method, the oil is methodically removed by hand, while in LM-CPOE, plastic bowls are employed for this purpose. The collected oil is then deposited into an aluminum pan until a thin oil layer remains atop the aqueous medium. The decanted material is squeezed by hand and the fibers and nuts are separated. In the case of LM-CPOE, the tank is equipped with a faucet for the convenient separation of water.

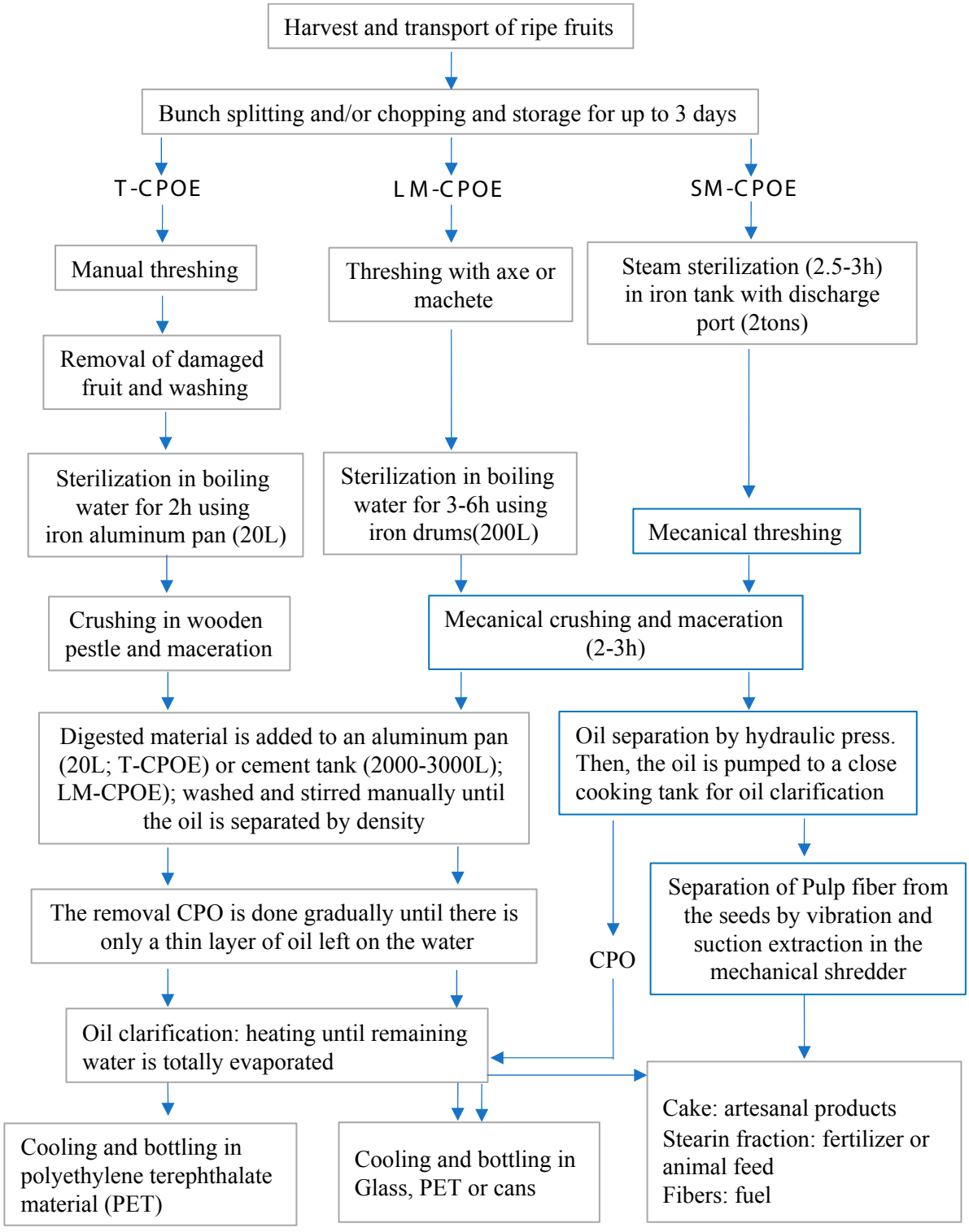

Figure 1. Schematic representation of the crude palm oil extraction (CPOE) methods. T-CPOE, traditional; LM-CPOE, little-mechanized; SM-CPOE, semi-mechanized.

In the SM-CPOE process, the oil is extracted by pressing. Thus, the digested fruit is conveyed by a screw conveyor (2.2 m x 50 cm x 50 cm) to a hydraulic press. After pressing, the extracted oil flows into a continuous tank (2 m x 1 m x 1 m) and then it is pumped into a nearby cooking tank (3 m x 1.6 m x 1.2 m) for oil clarification. The remaining intact seeds combined with pulp fibers (pulp cake) passes through a mechanical shredder (4 m x 90 cm x 20 cm). This shredder employs vibration and suction extraction to effectively separate the pulp fibers from the seeds.

The remaining water in the CPO is removed through a process known as oil clarification. This step essentially involves a drying process, where the oil is heated to facilitate the evaporation of water. Local processors employing the T-CPOE use 20 L aluminum pans for the oil clarification, whereas those using the LM-CPOE heat the oil in 200 L drums. It is worth noting that some T-CPOE producers introduce herbs, such as *Ocimum gratissimum L*., during this operation for flavor enhancement, primarily to mask off-flavors (Nwakodo *et al.*, 2020). The CPO is deemed adequately clarified when it ceases to boil. The duration of this process depends on factors such as the quantity of oil being clarified, and the efficiency of the wood used as fuel. Typically, the process takes between 1 and 2 h. The clarified oil is allowed to cool in 1 L glass or PET bottles (LM-CPOE) or 5 L cans (SM-CPOE) for commercialization. The dark orange-colored sludge, mixed with the fiber residues and settled at the bottom of the containers, is sometimes used in the production of soap or animal feed (Taiwo *et al.*, 2000). In the SM-CPOE, the oil is heated within a closed tank (6 m x 3 m x 1.5 m) for about 2-3 h. This tank features a small central opening at the top to facilitate water evaporation. Then the oil is pumped into storage tanks.

In all of these processes, the residual water generated in the sterilization and clarification stages is typically discarded and discharged onto the ground. On the other hand, the solid waste generated, including fibers, shells, and empty fruit bunches, is used as fuel in the boilers to generate the steam and energy required for the mill operation, or as animal feed (Robins, 2018,). The seeds are spread onto the ground adjacent to the mill and left to dry for approximately two weeks for later commercialization.

3.2. CPO characterization

The statistical analysis of the data did not show significant differences among the three collections throughout processing time, except for the DOBI and the C* color parameter when the LM-CPOE and SM-CPOE samples were respectively analyzed (Table 1). Consequently, the data from the various collections were analyzed as a whole to evaluate significant differences among the extraction processes.

The PV in the oils, ranging from 3.1 to 7.4 meq O_2/kg oil, was consistently below the recommended limit for CPO, as set by the Codex Alimentarius, 2023, which is 15 meq O_2/kg oil (Table 1). No significant differences in PV were found among the three extraction methods (Table 1). Ruswanto *et al*. (2020) demonstrated that fruit ripeness and storage duration can affect the PV, with oils from overripe fruit and prolonged storage showing higher values, even reaching 15 meq O2/kg. In the present study, the constant PVs throughout the season can be attributed to the use of the same fruit variety, sub spontaneous Dura (De Almeida *et al.*, 2013), and a standardized harvesting practice. Smallholders visually identify ripe FFBs based on specific criteria, including the color of detached fruitlets that have fallen to the ground (Lai *et al.*, 2023). Additionally, uniform fermentation periods (Figure 1) may contribute to the observed consistent PV.

The fermentation process is utilized to improve fruit loosening and decrease the time required for spikelet processing, which may result in the production of crude palm oil (CPO) with high free fatty acid (FFA) content. This could be avoided with adequate equipment for fruit processing. In this regard, the objective of this research is precisely to demonstrate the need for modernization of the CPO extraction industry in this region. The formation of peroxides in CPO often results from processing methods by which hot oil absorbs oxygen from the environment and the process is catalyzed by the presence of certain metals (e.g. iron) and steam. The values tend to be higher with longer periods of fermentation and storage.

The TC contents in the oils ranged between 375 and 598 ppm, with no significant differences observed among the extraction processes (Table 1). It is noteworthy that the Codex Alimentarius (2023) sets a broader range for TC values in CPO, ranging from

Table 1. Physicochemical parameters of CPO extracted by T-CPOE, LM-CPOE and SM-CPOE throughout the period between December 2017 and March 2018. Collections were performed at the beginning (1), at an intermediate time (2) and at the end of the period (3).

ANALYSIS	EXT. METHOD	COLLECTION			AVERAGE
		1	2	3	
PV (meq O_2/kg)	T-CPOE	3.9 ± 1.1^{aA}	3.8 ± 2.4^{aA}	3.1 ± 1.9^{aA}	3.6 ± 2.0^{a}
	LM-CPOE	7.6 ± 4.7^{aA}	5.7 ± 1.8^{aA}	4.1 ± 1.2^{aA}	5.8 ± 3.3^{a}
	SM-CPOE	3.2 ± 1.3^{aA}	5.1 ± 2.5^{aA}	5.0 ± 1.6^{aA}	4.5 ± 2.0^{a}
TC (ppm)	T-CPOE	466 ± 86^{aA}	597 ± 150^{bA}	519 ± 91^{aA}	528 ± 126^{a}
	LM-CPOE	374 ± 145^{aA}	450 ± 61^{aA}	495 ± 56^{aA}	440 ± 109^{a}
	SM-CPOE	443 ± 47^{aA}	465 ± 107^{abA}	474 ± 184^{aA}	461 ± 127^{a}
L*	T-CPOE	30.9 ± 0.8^{aA}	30.8 ± 0.8^{aA}	31.0 ± 1.2^{aA}	30.9 ± 0.9^{a}
	LM-CPOE	31.0 ± 1.0^{aA}	30.8 ± 0.7^{aA}	30.5 ± 0.5^{aA}	30.7 ± 0.8^{a}
	SM-CPOE	28.7 ± 1.9^{aA}	30.2 ± 1.2^{aA}	31.0 ± 1.0^{aA}	30.0 ± 1.7^{a}
a*	T-CPOE	11.3 ± 1.0^{aA}	12.7 ± 1.6^{aA}	11.9 ± 1.8^{aA}	11.9 ± 1.8^{a}
	LM-CPOE	9.6 ± 2.9^{aC}	11.7 ± 0.6^{aB}	12.3 ± 0.7^{aA}	11.2 ± 2.1^{a}
	SM-CPOE	9.8 ± 1.2^{aA}	11.2 ± 1.1^{aA}	11.4 ± 1.5^{baA}	10.8 ± 1.5^{a}
b*	T-CPOE	24.0 ± 1.9^{bA}	23.7 ± 1.4^{aA}	24.0 ± 2.1^{aA}	23.9 ± 1.8^{b}
	LM-CPOE	23.5 ± 2.0^{bA}	23.1 ± 1.2^{aA}	23.0 ± 0.7^{aA}	23.2 ± 1.4^{b}
	SM-CPOE	20.0 ± 2.9^{aA}	20.9 ± 4.7^{aA}	23.1 ± 2.0^{aA}	21.3 ± 3.1^{a}
C*	T-CPOE	26.5 ± 2.4^{aA}	27.1 ± 0.8^{bA}	26.8 ± 1.3^{aA}	26.8 ± 1.6^{b}
	LM-CPOE	25.5 ± 1.4^{aA}	25.9 ± 0.9^{abA}	26.1 ± 0.7^{aA}	25.8 ± 1.1^{b}
	SM-CPOE	22.3 ± 3.0^{aA}	24.4 ± 1.9^{aAB}	26.0 ± 0.7^{aB}	24.2 ± 2.6^{a}
$h_{ab}{}^{o}$	T-CPOE	65.0 ± 2.3^{aA}	61.7 ± 4.5^{aA}	63.4 ± 5.1^{aA}	63.4 ± 4.4^{a}
	LM-CPOE	67.5 ± 7.3^{aA}	63.2 ± 2.3^{aA}	61.9 ± 1.6^{aA}	64.2 ± 5.1^{a}
	SM-CPOE	63.6 ± 2.4^{aA}	62.8 ± 3.0^{aA}	61.9 ± 9.6^{aA}	62.8 ± 4.2^{a}
DOBI	T-CPOE	2.6 ± 0.8^{bA}	2.9 ± 0.8^{bA}	2.5 ± 0.8^{bA}	2.6 ± 0.8^{b}
	LM-CPOE	1.0 ± 0.4^{aA}	1.6 ± 0.2^{aB}	1.7 ± 0.4^{aB}	1.4 ± 0.5^{a}
	SM-CPOE	1.6 ± 0.4^{aA}	1.8 ± 0.3^{aA}	1.5 ± 0.4^{aA}	1.6 ± 0.4^{a}
OSI (h)	T-CPOE	2.5 ± 0.6^{aA}	3.3 ± 2.6^{aA}	2.5 ± 2.4^{aA}	2.8 ± 2.1^{b}
	LM-CPOE	1.5 ± 2.3^{aA}	0.6 ± 0.3^{aA}	0.7 ± 0.4^{aA}	1.4 ± 0.3^{a}
	SM-CPOE	3.3 ± 1.8^{aA}	1.9 ± 1.4^{aA}	2.6 ± 3.6^{aA}	2.5 ± 0.6^{b}

PV, peroxide value; DOBI, deterioration of bleachability index; TC, total carotenoids; L*, lightness; a*, negative values indicate green and positive values indicate red; b*, negative values indicate blue and positive values yellow; C*, chroma; $h_{ab}{}^{o}$, hue angle; OSI, oxidative stability index determined by the Rancimat test at 120 ºC. Results for each collection represent the mean and standard deviation of 4 (T-CPOE), 8 (LM-CPOE) and 7 (SM-CPOE) samples. Mean values of analytical triplicates were considered for each sample. Different lowercase letters indicate significant differences among the extraction processes for a given collection or considering the total set of data ($p < 0.05$) and different uppercase letters indicate significant differences among the collections for a given extraction process ($p < 0.05$) according to Duncan's test (homoscedasticity) or Games-Howell's test (heteroscedasticity).

500 to 2000 ppm. Comparatively, TC values reported for Bahia CPO have been in the vicinity of those of this study, with reports of 585 ppm (Feitosa *et al.*, 2019) and 545-578 ppm (De Almeida *et al.*, 2013). The relatively lower TC levels observed in Valença's oils may be attributed to potential losses of carotenes during processing. Factors such as high temperatures sustained for extended periods during sterilization and oil clarification, as well as light exposure during the clarification process, can contribute to the oxidation of carotenoids.

The CIELab coordinates did not reveal significant differences among the different CPO extraction processes (Table 1). All CPO samples were situated

within the first quadrant of the CIELab color space, showing positive values for the a* and b* parameters. However, slight but significant differences were observed among the extraction processes for the b* and C* parameters, with both being slightly lower for the SM-CPOE samples. It was observed that the hue values (h_{ab}^{o}) consistently placed the oils in the reddish-orange color zone (Table 1).

The DOBI is calculated as the ratio between the levels of TC and compounds that absorb UV light at 269 nm, specifically secondary lipid oxidation products containing conjugated triene structures (Basyuni *et al.*, 2017). Even though no significant differences were found in the TC contents among the three extraction processes, distinct variations were observed in the DOBI. Notably, the oils obtained through the T-CPOE method exhibited higher DOBIs, with values within the range of fair quality CPOs (2.31-2.92) (Table 1). Conversely, the DOBIs in the oils from the LM-CPOE and SM-CPOE processes were characteristic as poor-quality oils (1.68-2.30) (Gee, 1999). Several factors were observed across all processes that could have contributed to the reduction in TC levels and an increase in secondary oxidation products, consequently leading to low DOBI values (Basyuni *et al.*, 2017). These factors include delays in fruit processing, variations in sterilization time and temperature, contamination from sterilizer condensate water, exposure to badly oxidized sludge, and potential hygiene issues in the mills.

The OSI values for the oils were extremely low compared to reported values for CPOs produced in the same region (2.04-4.66 h) (De Almeida *et al.*, 2013). The OSI of the oils obtained by the LM-CPOE was lower ($p < 0.05$) compared to those obtained by the T-CPOE and SM-CPOE (Table 1). This clearly shows that the LM-CPOE oil had poor quality and suggests that the LM-CPOE method was the least suitable in terms of oil quality.

Similar to data in Table 1, no significant differences were found in the levels of TPC among the different collections, except for the polymer content when analyzing the LM-CPOE and SM-CPOE samples. Therefore, regardless of the collection, the data were analyzed to assess variances between methods. Across all CPO extraction processes, the levels of TPC ranged from 8.7 to 18.5 % (Table 2). Notably, the total contents of TPC were significantly lower in the T-CPOE samples (8.7-9.6%) compared to those found in the LM-CPOE (14.2-18.5%) and SM-CPOE (11.9-13.6%) oils (Table 2).

The total fraction of polar compounds in the CPOs primarily consisted of products from hydrolytic degradation, i.e. DAG and FFA. In descending order of concentration across all extraction methods, TPCs ranked as DAG ≈ FFA > oxTGM > (TGP + TGD) (Table 2). These results confirm the prevalence of DAG and FFA in fresh CPO (Nizam and Mahmud, 2021). DAG values for LM-CPOE and SM-CPOE were within those observed in the studies by Feitosa *et al*. (2019), who reported values of 8.7%. The DAG and FFA values were significantly lower in the oils obtained by the T-CPOE. The oils produced by the LM-CPOE and SM-CPOE methods can be regarded as inadequate CPOs because the FFA contents in the oils were higher than 5%, except the SM-CPOE sample in collection 3, which was slightly below, but still close to the limit (Codex Alimentarius, 2023).

The higher hydrolysis of the LM-CPOE and SM-CPOE samples is related to the processing of damaged fruits, in which enzymatic hydrolytic reactions are initiated by the contact of natural lipases with the oil (Ruswanto *et al.*, 2020). The meticulous fruit handling practiced in the T-CPOE process is crucial in preventing hydrolysis in CPO and ensuring the production of high-quality oils. Additionally, the absence of equipment made from iron material in this process further contributes positively. Thermal and oxidative reactions take place during oil extraction due to the high temperatures employed, as well as to the presence of polyunsaturated fatty acids (linoleic) that produce oxTGMs and, in advanced stages of oxidation, polymerized compounds (TGPs). Basically, the conversion of TAG to oxTGM is considered the beginning of oxidation. Similar to PV, no significant differences were observed in the levels of oxTGM among the three extraction methods (Table 2). Feitosa *et al*. (2019) detected 1.3% of oxTGM for CPO produced in Valença. CPO extraction includes several heating steps, which give rise to the formation of hydroperoxides (primary oxidation compounds) and aldehydes and ketones, among other secondary oxidation products. Both primary and secondary oxidation products form the group of oxidized triacylglycerols (oxTGM). The significant differences found in the DOBI between the T-CPOE and the other two extraction processes (Table 1) were not observed for the oxTGM (Table 2). The TPC analysis is an

Table 2. Total and individual contents of polar compounds in CPO extracted by T-CPOE, LM-CPOE and SM-CPOE throughout the period between December 2017 and March 2018. Collections were performed at the beginning (1), at an intermediate time (2) and at the end of the period (3).

ANALYSIS	EXT. METHOD	COLLECTION			AVERAGE
		1	2	3	
TPC (%)	T-CPOE	8.7 ± 1.6^{aA}	9.5 ± 1.2^{aA}	9.6 ± 2.5^{aA}	9.3 ± 1.8^{a}
	LM-CPOE	18.5 ± 6.5^{bA}	15.6 ± 4.0^{bA}	14.2 ± 2.4^{bA}	16.1 ± 4.4^{c}
	SM-CPOE	13.6 ± 2.8^{abA}	13.7 ± 5.5^{bA}	11.9 ± 2.5^{abA}	13.1 ± 3.0^{b}
TGP +TGD (%)	T-CPOE	0.2 ± 0.0^{aA}	0.2 ± 0.1^{aA}	0.1 ± 0.1^{aA}	0.1 ± 0.1^{a}
	LM-CPOE	0.5 ± 0.1^{cB}	0.3 ± 0.1^{bA}	0.2 ± 0.1^{aA}	0.3 ± 0.1^{c}
	SM-CPOE	0.3 ± 0.1^{bB}	0.2 ± 0.1^{aA}	0.2 ± 0.0^{aB}	0.2 ± 0.1^{b}
oxTGM (%)	T-CPOE	0.7 ± 0.4^{aA}	0.6 ± 0.2^{aA}	0.6 ± 0.2^{aA}	0.7 ± 0.3^{a}
	LM-CPOE	2.0 ± 2.6^{aA}	0.8 ± 0.2^{aA}	0.8 ± 0.2^{aA}	1.2 ± 1.5^{a}
	SM-CPOE	0.9 ± 0.2^{aA}	0.9 ± 0.4^{aA}	0.8 ± 0.2^{aA}	0.9 ± 0.3^{a}
DAG (%)	T-CPOE	4.8 ± 0.9^{aA}	5.1 ± 0.6^{aA}	5.3 ± 1.6^{aA}	5.1 ± 1.0^{a}
	LM-CPOE	7.4 ± 1.7^{bA}	7.2 ± 1.1^{bA}	7.4 ± 1.3^{bA}	7.3 ± 1.4^{b}
	SM-CPOE	6.4 ± 1.7^{abA}	6.6 ± 1.6^{abA}	6.6 ± 1.6^{abA}	6.6 ± 1.6^{b}
FFA (%)	T-CPOE	3.6 ± 1.1^{aA}	3.6 ± 0.7^{aA}	3.2 ± 1.1^{aA}	3.5 ± 0.9^{a}
	LM-CPOE	8.0 ± 2.5^{bA}	7.9 ± 1.4^{bA}	6.0 ± 1.9^{bA}	7.3 ± 2.1^{c}
	SM-CPOE	5.9 ± 1.4^{abA}	5.4 ± 2.3^{aA}	4.6 ± 1.5^{abA}	5.3 ± 1.8^{b}

T-CPOE, traditional crude palm oil extraction; LM-CPOE, little mechanized crude palm oil extraction; SM-CPOE, semi-mechanized crude palm oil extraction; TPC, total polar compounds; TGP, triacylglycerol polymers; TGD, triacylglycerol dimers; oxTGM, oxidized triacylglycerol monomers; DAG, diacylglycerols; FFA, free fatty acids. Results for each collection represent the mean and standard deviation of 4 (T-CPOE), 8 (LM-CPOE) and 7 (SM-CPOE) samples. Mean values of analytical duplicates were considered for each sample. Different lowercase letters indicate significant differences among the extraction processes for a given collection or considering the total set of data ($p < 0.05$) and different uppercase letters indicate significant differences among the collections for a given extraction process ($p < 0.05$) according to Duncan's test (homoscedasticity) or Games-Howell's test (heteroscedasticity).

accurate method to evaluate used frying oils, which normally present considerably high thermoxidative degradation. When applied to oils that have not been heated or oils with low oxidation, the quantification of oxTGM is subject to a considerably high error due to incomplete separation between the non-polar and polar fractions. It is very common that trace levels of the non-polar triacylglycerols are not completely separate and they are collected in the polar fraction. Even when these levels are very low, they contribute significantly to increasing the low levels of oxTGM, as they (TAG and oxTGM) coelute and so are determined together in the HPSEC analysis. In used frying oils, which normally comprise relatively high levels of oxTGM, the contribution of non-separated TAG is negligible.

The levels of polymers found in the present study were, as expected, very low (0.1-0.5%) (Table 2) and in agreement with those reported by Khor *et al*. (2019), who reported 0.5% in palm oil and 0.1-0.8% in pure olein (Table 2). Triacylglycerol polymers (TGP+TGD) are indicative of thermal degradation at elevated temperatures. Sterilization and oil clarification are thermal treatments in the CPO extraction processes. However, the temperature does not surpass the water boiling temperature (100 ºC) in the sterilization step. In contrast, if the time taken in the oil clarification is not well controlled, temperatures higher than 100 ºC can be reached after water evaporation, resulting in polymerization (Feitosa *et al.*, 2019).

3.3. Linear correlations between the physicochemical parameters

The entire dataset obtained in this study was analyzed to examine possible linear correlations between the different physicochemical parameters. As expected, a negative correlation was observed between the DOBI and the oxidation indicators, namely, DOBI with TPC (r = -0.634, $p < 0.01$), TGP+TGD (-0.755, $p < 0.01$) and oxTGM (-0.439; $p < 0.01$) (Table 3). Additionally, a positive correlation was found between DOBI and TC (0.634 $p < 0.01$). The decrease in DOBI indicates a reduction in the levels of carotenoids and/or an increase in oxidation. Similar to the DOBI, an inverse correlation was observed between carotenoids and the oxidation compounds, i.e. (TGP +TGD) (r = -0.347; $p < 0.01$) and the PV (r = -0.366; $p < 0.01$) (Table 3), which could partially explain the lower TC contents found in the oils. An inverse correlation of TC with L* (r = -0.514, $p < 0.05$), b= (r = - 0.482, $p < 0.01$) and $h_{ab}{}^{o}$ = (r = -0.840, $p < 0.01$) was also observed, and a positive correlation with a* (0.811; $p < 0.01$) (Table 3), indicating that the lower the carotenoid content, the more transparent, the less orange and the more yellow the CPO becomes. De Almeida *et al*. (2018) showed changes in the color of CPO samples stored at 26-32 °C and 20-25 °C after 9 months of storage with increases in L*, b* and h_{ab} and a decrease in a*, indicating that oxidation led to a loss in orange color in the oils.

Positive correlations were found among oxTGM, DAG, FFA and PV (Table 3). High concentrations of

Table 3. Spearman's correlation coefficients between the physicochemical parameters analyzed in this study considering the entire dataset.

	TGP+TGD (%)	oxTGM (%)	DAG (%)	FFA (%)	PV meq O_2/kg	OSI_{120} (h)	TC (mg/kg)	DOBI
TPC (%)	0.636***	0.481***	0.837***	0.896***	0.260**	-0.575***	-0.223*	-0.634***
TGP+TGD (%)		0.619***	0.481***	0.542***	0.253*	-0.380***	-0.347***	-0.755***
oxTGM (%)			0.352***	0.398***	0.316**	-0.205	-0.212	-0.439***
DAG (%)				0.675***	0.278**	-0.621***	-0.217*	-0.514***
FFA (%)					0.166	-0.458***	-0.103	-0.526***
PV (mEq kg^{-1})						-0.424***	-0.366***	-0.403**
OSI_{120} (h)							0.422***	0.531***
L*							-0.514**	-0.323*
a*							0.811***	0.491**
b*							-0.482***	-0.191
C*							-0.133	0.074
$h_{ab}{}^{o}$							-0.840***	-0.431**
TC (mg/kg)								0.634***

TGP, triacylglycerol polymers; TGD, triacylglycerol dimers; oxTGM, oxidized triacylglycerol monomers; DAG, diacylglycerols; FFA, free fatty acids; PV, peroxide value; OSI, oxidative stability index; TC, total carotenoids; DOBI, deterioration of bleachability index; L*, lightness; a* and b*, color parameters representing, respectively, the green-red and blue-yellow colors; C*, chroma; $h_{ab}{}^{o}$, hue angle. Asterisks denote significant correlations at 0.1 (*), 0.05 (**) or 0.01 (***) confidence level

FFA in CPO indicate a deterioration of oil quality, as these compounds are known to promote oxidative reactions. Notably, TPC, TGP+TGD and the DOBI were correlated with the FFA content, regardless of the extraction conditions, as indicated by significant ($p < 0.05$) Sperman's correlation coefficients of 0.896, 0.542 and -0.526, respectively (Table 3).

4. CONCLUSIONS

The oils obtained by the T-CPOE method exhibited superior quality compared to those produced by the LM-CPOE and SM-CPOE methods, even though the latter were methods with a higher degree of mechanization contributing to increased yield. The selection of fresh and undamaged fruits, along with the practices used in the T-CPOE, are crucial factors that significantly reduce oil degradation (diacylglycerols, free fatty acids, total polar compounds). Although the LM-CPOE and SM-CPOE methods involved less manual interference, the absence of good manufacturing practices necessitates technical modifications for both methods. Modernizing the production process, implementing good manufacturing practices, providing training to producers, and offering government incentives can be strategic measurements to enhance and ensure the quality and nutritional properties of CPO.

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DECLARATION OF COMPETING INTEREST

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AUTHORSHIP CONTRIBUTION STATEMENT

R.S. Bomfim: Investigation, Methodology, Writing – original draft. **J. Velasco:** Writing – review & editing, Methodology, Data Curation. **L.A. Cardoso:** Methodology, Writing – review & editing; **C.D.F. Ribeiro:** Methodology, Writing – review & editing; **L.Q.M. Marinho:** Methodology, Writing – review & editing; **P.R. Ribeiro**: Methodology. **D.T. de Almeida:** Conceptualization, Supervision, Writing - review & editing, Visualization.

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