Effects of Heating and Treatment Time on Some Characteristics of *Terminalia catappa* Seeds Oils Harvested in Côte d'Ivoire

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Abstract— Effects of heating on some quality characteristics of Terminalia catappa seeds oils from unripe and ripe mature fruits has been investigated using standard analytical methods system. Peroxide (PV), acid (AV), iodine (IV) and saponification (SP) value, absorptivity at 232 and 270 nm, and refractive index (RI) seed oil were less changed. The two studied Terminalia catappa seed oils presented high oxidation stability. They were resistant to thermal treatment (150 - 275 °C) during a long period (~480-120 min.) regarding the high stability of some of their quality parameters. This may indicate that they could bear thermal treatments that could be applied in refining procedures or in culinary treatments such as frying and cooking conditions. We could also expect that they may have a good shelf life.

Index Terms— Côte d'Ivoire, Heat treatment, oils, quality characteristics, *Terminalia catappa* unripe and ripe seeds.

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1 Introduction

Terminalia catappa tree belongs to the Combretaceae family, with a Meridional Asia origin [1]. It is a large, spreading tree distributed throughout the tropics in coastal environments. The tree grows principally in freely drained, well aerated, and sandy soils. It is widely planted for shade, ornamental purposes, and edible nuts [2]. In fact, there is hardly any public quarter where the tree is not found in Abidjan (Côte d'Ivoire). Several species of the genus Terminalia have long been used in the traditional medicine in both East and West African countries to treat infectious diseases [3]. In Asian countries, the extract of the leaves has been used in folk medicine for treating dermatitis and hepatitis [4]. It has shown anti-oxidative, anti-inflammatory and hepatoprotective actions [5]. Several tannins of this extract have shown inhibiting HIV replication in infected H9 lymphocytes with little cytotoxicity [6]. In addition, Terminalia catappa (T C) fruit contains a very hard kernel with an edible almond [7]. The nuts may be consumed fresh shortly after extraction from the shell or else preserved by smoking. In some areas, the nuts are mainly a snack food consumed by children, with the fleshy fruit also sometimes being consumed. In other areas, tropical almond nuts are highly regarded as a human food source. Proximate analyse of Terminalia catappa seed shows that it has high amounts of protein and oil [8]. According to Abdullahi and Anneli [9], Ajayi et al. [10] and dos Santos et al. [2] the fatty acid composition of Terminalia catappa seed oil shows high amount of unsaturated fatty acids with linoleic and oleic acids as the major ones. dos Santos et al. [2] and Monnet et al. [11] reported studies concerning chemical and physical characteristics of these oils.

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Nearly, Janporn et al. [12] showed that physicochemical properties including the density, refractive index, melting point, acidity, free fatty acid, saponification value, unsaponifiable, peroxide, and fatty acid composition of the extracted oil were comparable with soybean oil and their values followed the dietary standard of edible oil. The seed oils of mature Terminalia catappa ripe and unripe fruits from Côte d'Ivoire were analysed for their main chemical compositions. These seed oils had characteristically low acidity and peroxide values and showed some absorbance in the UV-B and UV-C ranges. Unripe and ripe seed oils originated from Côte d'Ivoire were a good quality and could be used in cosmetic, pharmaceutical and food products.
The use of Terminalia catappa seed oil for industrial applications or for culinary preparations could necessitate its exposure to high temperature such as refining operation, frying, cooking conditions, etc. These thermal treatments could lead to changes in quality characteristics of the oils.
The aim of this present work was to study physicochemical changes of Terminalia catappa seed oils during heating. Following heat-induced physicochemical characteristics changes of Terminalia catappa seed oils, we could also indicate until what stage they could bear this treatment.

2 Material and Methods

2.1 Materials

Mature fruits of TC were harvested from the trees at the University of Nangui Abrogoua (Abidjan, Côte d'Ivoire). The climate in this area is characterised by high humidity, precipitation up to 4,000 mm per year and relatively high temperatures, averaging 28°C. All the chemicals, reagents and solvents used in the experiments were of analytical grade and were products of Sigma Chemical Co. (St. Louis, MO).

2.2 Extraction and physicochemical characteristics of Terminalia catappa oil seeds

The oil was extracted using hexan as solvent in Soxhlet apparatus. The solvent was removed by rotary vacuum evaporator and the oil was stored in refrigerator (4 °C) for following experiments. The oil seed stability was investigated at 150 and 275°C after exposure to each temperature for a period of 30 to 480 min. Standards ISO (International Organisation for Standardisation) were used for the determination of the peroxide value (ISO, 3960), acidity (percentage of free fatty acid was calculated as oleic acid) (ISO, 660), iodine value (ISO, 3596) and saponification value (ISO, 3657) of oil. Refractive index was determined by AOAC method 41.1.07 [13] using an Abbe' refractometer (Bausch and Lomb, Salt Lake, UT). The viscosity was followed at 25°C with a Stress Tech Rheologica Rheometer (Rheologica Instruments AB, Lund, Sweden) conducted with a steel cone-plate (C40/4) under a constant shear rate of 100 s-1 according to Besbes et al. [14] method.

Analyses of fatty acid methyl esters were carried out with a Hewlett Packard Gas Chromatograph (Model, 439), equipped with a hydrogen flame ionisation detector and a capillary column, Supelcowax Tm, fused silica (60 m x 0.25 mm id., of 0.2 µm particle diameter). Temperatures of injector, column and detector were 260, 180 and 250°C, respectively. Hydrogen was used as carrier gas at a flow rate of 30 ml/min. Identification and quantification of fatty acid methyl esters was accomplished by comparing the retention times of the peaks with those of standards. Absorptivity of oil solutions (1%, v/v) in hexane were measured using a spectrophotometer (JASCO V-530, WITEG Labortechnik., Gmbh).

Heat process

Heat process was done using a domestic electronic heater plate with an intelligent magnetic stirrer and the samples were heated at 150 °C and 275 °C for 30 to 480 min. Aliquots were drawn at intervals and immediately cooled in a freezer for subsequent analyses. Experiments were performed in triplicate.

Statistical analyses

The mean values and standard deviations of each analysis are reported. Analysis of variance (ANOVA) was performed as part of the data analyses (SAS, 1988). When F-values were significant ($p<0.05$) in ANOVA, then least significant differences were calculated to compare treatment means.

3 Results and Discussion

Chemicals parameters at 150 and 275 °C

The changes in peroxide, acid, iodine, and saponification value of the oils used in this heat treatment experiment are shown in tables 1 and 2.

The results show a gradual increase of acid value (AV) for Terminalia catappa unripe and ripe seeds oils throughout the heat treatments at 150 and 275 °C. Higher acid values in the two heated oils were observed at 275 °C throughout the first 4 hours and beyond this time at 150 °C. However, the AV of the unripe and ripe seeds oils heated at 150 °C and 275 °C did not exceeded 4 mg KOH/g of oil. The increase in the acidity of these oils can be explained by the hydrolysis of ester-bound glycerides and phospholipids in order to release fatty acids, but also by the cleavage of the double bonds of the fatty acids which are already free under the oxidation phenomena in order to release diacids in the same medium [15, 16]. Despite this physical action, the AV of Terminalia catappa seeds oils obtained during heating at 150 and 275 °C for 480 minutes remain below 5. Thus, according to Codex Alimentarius [17], these substances are in good quality.

Saponification value (SV) for Terminalia catappa unripe and ripe seeds oils obtained remain constant besides 120 minutes of heat treatments at 150 and 275 °C. Beyond this processing time, the measured values at 275 °C were observed to be the highest (240 mg KOH/oil). But, at 150 °C this did not reach 210 mg KOH/oil after 480 minutes. These values are within the range recommended by FAO/WHO [18] international standard for edible oil which is 181 ± 2.60. The lower SV suggests that the mean molecular weight of fatty acids is lower or that the number of ester bonds is less. This might imply that the fat molecules did not interact with each other [19]. The iodine value (IV) is a measure of the unsaturated acid present in the oil. Therefore, the test measures the amount of iodine consumed by the acid. The iodine value decreases in a uniform manner with an increase in temperature. Thus, sample heated at 275 °C has the lowest iodine value, indicating that the higher the temperature, the lower the iodine value. This agreed with the finding [20] that there is decreasing trend in iodine value of the oil during deep-fat frying. The decrease in IV with time of frying could be attributed to the changes in fatty acids taking place with duration of frying [21]. A decrease in IV is an indicator of lipid oxidation [22] and is consistent with the decrease in double bonds as oil becomes oxidized [23]. Despite the heating of these oils, the IV does not exceed 70 g/100 g oil.

The peroxide value (PV) is expressed in mEq O2/kg, it is formed as a result of oxidation of oil. It was observed that these oils are only a minor resistant to oxidation in high temperature (275 °C). The oil is considered rancid at a peroxide value above 10 mEq O2/kg [24]. The rise in peroxide formation in Terminalia catappa unripe and ripe seeds oils reach to average oxidation state right after 120 minutes of heating at 275 °C. But, PV remains under average oxidation state after 480 minutes of heating at 150 °C. Whereas storage of oil leads to increases in PV, the use of oils for frying does not lead to substantial increases in PV because peroxides decompose spontaneously above 150 °C [25]. According to Codex Alimentarius Commission [17], these substances are edible vegetable oils. These are oils of excellent qualities.

Physical parameters at 150 and 275 °C

Absorptivity in the UV range could be a good tool for the analysis of oxidized oils. The formation of hydroperoxides is coincidental with conjugation of double bonds in polyunsaturated fatty acids, measured by absorptivity in the UV spectrum [26].

TABLE 1. EVOLUTION OF CHEMICAL PARAMETERS OF UNRIPE (USO) AND RIPE (RSO) SEEDS OIL OF *TERMINALIA CATAPPA* DURING HEAT TREATMENTS AT 150 °C DURING TIME

Treatment time (min)	Chemical parameters: Peroxide index (mEqO2 / kg oil)		Acid index (mg KOH / g oil)		Saponification value (mg KOH / 100 g oil)		Iodine index (g / 100 g oil)	
	RSO	USO	RSO	USO	RSO	USO	RSO	USO
30	0.40 ± 0.0a	0.49 ± 0.01a	0.80 ± 0.0a	0.75 ± 0.05a	189.54 ± 0.11a	189.90 ± 0.13a	70.16 ± 0.121	70.18 ± 0.211
60	1.27 ± 0.11b	1.49 ± 0.03b	1.53 ± 0.0c	1.59 ± 0.06c	192.35 ± 0.25b	197.74 ± 0.35cd	69.53 ± 0.271	68.65 ± 0.20k
120	2.80 ± 0.17c	2.51 ± 0.43c	1.94 ± 0.0d	1.98 ± 0.01d	196.89 ± 0.03c	201.36 ± 0.45gh	67.68 ± 0.06ij	67.43 ± 0.07i
240	6.65 ± 0.34g	5.57 ± 0.51f	2.43 ± 0.0f	2.47 ± 0.15f	200.05 ± 0.04efg	202.46 ± 0.48h	65.44 ± 0.41fg	65.23 ± 0.12fg
360	8.48 ± 0.40ij	7.83 ± 0.29h	3.18 ± 0.0j	3.26 ± 0.04j	208.71 ± 0.29j	205.91 ± 0.14i	65.08 ± 0.50fg	64.58 ± 0.36f
480	8.84 ± 0.04jk	9.02 ± 0.03k	3.93 ± 0.0k	4.14 ± 0.031	212.29 ± 0.35k	214.0 ± 0.051	61.81 ± 0.09d	61.42 ± 0.25d

RSO: Ripe Seeds Oils
USO: Unripe Seeds Oils

TABLE 2. EVOLUTION OF CHEMICAL PARAMETERS OF UNRIPE (USO) AND RIPE (RSO) SEEDS OIL OF *TERMINALIA CATAPPA* DURING HEAT TREATMENTS AT 275 °C DURING TIME

Treatment time (min)	Chemical parameters: Peroxide index (mEqO_2 / kg oil)		Acid index (% as oleic acid)		Saponification value (mg KOH / g oil)	
	RSO	USO	RSO	USO	RSO	USO
30	4.08 ± 0.04^{d}	4.79 ± 0.26^{e}	1.16 ± 0.06^{b}	1.09 ± 0.01^{b}	198.35 ± 0.04^{cde}	198.17 ± 0.14^{cd}
60	8.62 ± 0.11^{jk}	8.11 ± 0.32^{hi}	2.15 ± 0.04^{e}	2.00 ± 0.11^{d}	198.95 ± 0.01^{def}	198.93 ± 0.03^{def}
120	9.83 ± 0.01^{l}	10.05 ± 0.04^{l}	1.98 ± 0.02^{d}	2.18± 0.18^{e}	200.44 ± 0.03^{fg}	201.16 ± 0.36^{gh}
240	13.11 ± 0.12^{m}	13.09 ± 0.27^{m}	2.64 ± 0.00^{g}	2.74 ± 0.20^{g,h}	233.82 ± 0.10^{n}	226.12 ± 0.34^{m}
360	15.26 ± 0.43^{n}	15.82 ± 0.16^{o}	2.85 ± 0.04^{h}	2.99 ± 0.10^{i}	238.01 ± 0.00^{o}	233.01 ± 0.00^{n}
480	16.05 ± 0.04^{o}	16.54 ± 0.10^{p}	3.31 ± 0.02^{j}	3.31 ± 0.02^{j}	240.54 ± 0.16^{p}	242.38 ± 0.51^{q}

RSO: Ripe Seeds Oils
USO: Unripe Seeds Oils

Tables 3 and 4 illustrate the evolution of the absorptivity at 232 nm and at 270 nm, over the range time 0 – 480 minutes
It was observed slightly increase at 150 °C (respectively 3 to 3.7 and 0.7 to 1.3) throughout 480 minutes. However, the higher absorptivity was at 275 °C (respectively 3.25 to 3.8 and 1.4 to 1.6). Indeed, auto-oxidation products of fats have characteristic spectra in the ultraviolet. Thus, hydroperoxides of linoleic and oxidized fatty acids such as dienes resulting from peroxides decomposition, have an absorption spectrum around 232 nm [27]. The primary products of oxidation are not stable under heating and then their degradation could promote the formation of secondary product of oxidation that absorb at about 270 nm [28]. Low change in absorptivity at 232 nm and at 275 °C in Terminalia catappa seeds oils, regardless of fruits ripening are influenced by temperature and heating time. The slight increase in oil absorptivity is probably due to the low presence of peroxides or oxidized acids in these oils.

Auto-oxidation depends on several factors, including the degree of oil unsaturation, the free fatty acids, the presence of metallic traces and water, the packaging used, the ambient temperature and atmospheric oxygen. In contrast, photo oxidation is affected by oil components, as total amount of chlorophyll pigments and natural antioxidants (beta carotene, tocopherols and phenols).

One of the parameters used to determine the physical changes of the frying oils was the refractive index (RI). RI of both oils does not practically change during 480 minutes under different heating temperatures (Table 3 and 4). This could also confirm Terminalia catappa seeds oils have a good resistance against heating.

TABLE 3. EVOLUTION OF PHYSICAL PARAMETERS OF UNRIPE (USO) AND RIPE (RSO) SEEDS OIL OF *TERMINALIA CATAPPA* DURING HEAT TREATMENTS AT 150 ° C DURING TIME

Treatment time (min)	Physical parameters					
	Absorption coefficient K232		Absorption coefficient K270		Refractive index	
	RSO	USO	RSO	USO	RSO	USO
30	3.07 ± 0.00a	3.09 ± 0.02ab	0.70 ± 0.00b	0.70 ± 0.01b	1.46 ± 0.00fg	1.46 ± 0.00fg
60	3.07 ± 0.00a	3.11 ± 0.05b	0.72 ± 0.00b	0.72 ± 0.02b	1.45 ± 0.00efg	1.45 ± 0.00efg
120	3.27± 0.03cd	3.28 ± 0.01cd	0.82 ± 0.01c	0.82 ± 0.01c	1.45 ± 0.00defg	1.46 ± 0.00defg
240	3.30 ± 0.00cd	3.31 ± 0.01de	0.64 ± 0.02a	0.72 ± 0.02b	1.45 ± 0.01cdefg	1.45 ± 0.01cdefg
360	3.34 ± 0.00ef	3.38 ± 0.05f	1.03 ± 0.00d	1.12 ± 0.04e	1.45 ± 0.01cdef	1.45 ± 0.01cdef
480	3.68 ± 0.00kl	3.71 ± 0.01l	1.20 ± 0.01f	1.29 ± 0.02g	1.45 ± 0.00bcd	1.44 ± 0.00bc

RSO: Ripe Seeds Oils
USO: Unripe Seeds Oils

TABLE 4. EVOLUTION OF PHYSICAL PARAMETERS OF UNRIPE (USO) AND RIPE (RSO) SEEDS OIL OF *TERMINALIA CATAPPA* DURING HEAT TREATMENTS AT 275 °C DURING TIME

Treatment time (min)	Physical parameters					
	Absorbance coefficient K232		Absorbance coefficient K270		Refractive index	
	RSO	USO	RSO	USO	RSO	USO
30	3.25 ± 0.04c	3.35 ± 0.03f	1.40 ± 0.01h	1.48 ± 0.02ij	1.45 ± 0.00fg	1.45 ± 0.00fg
60	3.46 ± 0.00g	3.54 ± 0.05h	1.45 ± 0.00i	1.49 ± 0.01m	1.46 ± 0.00g	1.45 ± 0.00fg
120	3.55 ± 0.03h	3.58 ± 0.01h	1.47 ± 0.01i	1.52 ± 0.01jk	1.46 ± 0.00fg	1.46 ± 0.00defg
240	3.63 ± 0.00i	3.63 ± 0.01ij	1.48 ± 0.00ij	1.55 ± 0.01k	1.45 ± 0.01defg	1.45 ± 0.01defg
360	3.64 ± 0.00ijk	3.67 ± 0.02jkl	1.49 ± 0.0ij	1.55 ± 0.07k	1.45 ± 0.01ab	1.45 ± 0.01bcde
480	3.80 ± 0.01m	3.84 ± 0.03n	1.50 ± 0.00ij	1.62 ± 0.01l	1.44 ± 0.00a	1.44 ± 0.00ab

RSO: Ripe Seeds Oils
USO: Unripe Seeds Oils

CONCLUSION

The two studied *Terminalia catappa* seed oils presented high oxidation stability. Results show that both Terminalia catappa seed oils studied could resist thermal treatments that may be applied during frying, cooking conditions or during refining processes. Terminalia catappa seed oils were resistant to thermal treatment during a long period (~480-120 min.) regarding the high stability of some of their quality parameters. So, we could predict that they may have a good shelf-life and then could be stored safely during a long period. This hypothesis will be supported by the study of the behaviour of Terminalia catappa seeds oils during storage at ambient temperature.

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