Rev Esp Nutr Hum Diet. 2018; **22(1)**: 52 - 63

Revista Española de
Nutrición Humana y Dietética
Spanish Journal of Human Nutrition and Dietetics

www.**renhyd.org**

RESEARCH ARTICLE

Determining the oxidative stability and quality of tiger nut (*Cyperus esculentus*) oil and its antioxidant activity during microwave heating

Atoosa Sobhani[a], **Abdulkarim Sabo Mohammed**[a,*], **Fatemeh Ghobakhlou**[a], **Hasanah Mohd Ghazali**[a]

[a] Department of Food Science, Faculty of Food Science and Technology, Universiti Putra Malaysia, Serdang, Selangor, Malaysia.

[*] **karimsabo@upm.edu.my**

Editor Assigned: Eduard Baladia. Comité Editorial de la Revista Española de Nutrición Humana y Dietética. Pamplona, España.

Received: 17/05/2017; accepted: 11/03/2018; published: 16/03/2018.

Determining the oxidative stability and quality of tiger nut (*Cyperus esculentus*) oil and its antioxidant activity during microwave heating

ABSTRACT

Introduction: The emphasis on high-oleic vegetable oils is prominent in human communities all over the world. That being said, the high level of monounsaturated fatty acid (oleic acid) in tiger nut (*Cyperus esculentus*) oil shows that it is resistant to oxidative stability. The purpose of this study, therefore, was to see if tiger nut oil can be exploited for use as an alternative or supplementary source of high-quality and nutritious cooking oil.

Material and Methods: In this study, Color, RI, viscosity, PV, p-AV, FFA, TPC, $E^{1\%}_{1cm}$ at 233 and 269nm, thermal behavior, TAG and FAC were used to evaluate the oil after microwave heating.

Results: The PV, p-AV, FFA, TPC and specific extinction were increased during the microwave heating. Significant differences ($p<0.05$) were detected for peroxide, anisidine, acid value, polar compounds and specific extinction. During microwave heating, the amounts of peroxide, anisidine and TOTOX values increased from initial value 3.06, 0.72 and 6.84 for unheated oil to 4.11, 10.02 and 18.25 after 15 minutes heating respectively. Free fatty acid changed from 0.10 to 0.12% during microwave heating. Amount of unsaturated fatty acids decreased during the heating significantly. During microwave heating the antioxidant activity was significantly decreased ($p<0.05$) from 68.60 to 19.66 (for unheated tiger nut oil and after 15 minutes heating in high concentrations by DPPH test, respectively).

Conclusions: This may indicate that it can bear thermal treatments in such culinary methods as frying, and it can thus be concluded that tiger nut oil is stable in heating processes, especially frying.

KEYWORDS

Cyperus;

Oxidation-Reduction;

Microwaves;

Antioxidants.

doi: 10.14306/renhyd.22.1.407

Determinación de la estabilidad oxidativa y la calidad del aceite de chufa (*Cyperus esculentus*) y su actividad antioxidante durante el calentamiento por microondas

PALABRAS CLAVE

Cyperus;

Oxidación-Reducción;

Microondas;

Antioxidantes.

RESUMEN

Introducción: El énfasis en los aceites vegetales con alto contenido de ácido oleico es prominente en las comunidades humanas de todo el mundo. Dicho esto, el alto nivel de este ácido graso monoinsaturado (ácido oleico) en el aceite de chufa (*Cyperus esculentus*) muestra que es resistente a la estabilidad oxidativa. El propósito de este estudio fue ver si el aceite de chufa puede explotarse para su uso como una fuente alternativa o suplementaria de aceite de cocina de alta calidad y nutritivo.

Material y Métodos: En este estudio se utilizó el Color, RI, viscosidad, PV, p-AV, FFA, TPC, $E^{1\%}_{1cm}$ a 233 y 269nm, comportamiento térmico, TAG y FAC para evaluar el aceite después del calentamiento por microondas.

Resultados: El PV, p-AV, FFA, TPC y extinción específica aumentaron durante el calentamiento con microondas. Se detectaron diferencias significativas (p<0,05) para el peróxido, la anisidina, el valor del ácido, los compuestos polares y la extinción específica. Durante el calentamiento con microondas, las cantidades de los valores de peróxido, anisidina y TOTOX aumentaron desde el valor inicial de 3,06, 0,72 y 6,84 para el aceite no calentado a 4,11, 10,02 y 18,25 después de 15 minutos de calentamiento respectivamente. El ácido graso libre cambió de 0,10 a 0,12% durante el calentamiento con microondas. La cantidad de ácidos grasos insaturados disminuyó significativamente durante el calentamiento. Durante el calentamiento con microondas, la actividad antioxidante disminuyó significativamente (p<0,05) de 68,60 a 19,66 (para el aceite de chufa sin calentar y después de 15 minutos de calentamiento en altas concentraciones mediante la prueba de DPPH, respectivamente).

Conclusiones: Esto puede indicar que el aceite de chufa puede soportar tratamientos térmicos en métodos culinarios como la fritura y se puede concluir que el aceite de chufa es estable en los procesos de calentamiento, especialmente la fritura.

CITATION

Sobhani A, Abdulkarim SM, Ghobakhlou F, Ghazali HM. Determining the oxidative stability and quality of tiger nut (*Cyperus esculentus*) oil and its antioxidant activity during microwave heating. Rev Esp Nutr Hum Diet. 2018; 22(1): 52-63. doi: 10.14306/renhyd.22.1.407

INTRODUCTION

The tiger nut plant belongs to the sedge family, and the tiger nut itself is a tuberous rhizome that protrudes from the tips of the plant's roots under the ground. The aforementioned sedge family, or Cyperaceae, is a family of monocotyledonous plants which include up to 4000 species. That being said, the tiger nut is spherical and edible. The plant grows typically in the northern and mid belt regions of Nigeria, although it is also widely grown in wet places as a type of grass[1] .The tiger nut, which is actually a tuber, is valuable as a source of glucose, potassium and phosphorus as well as vitamins C and E. More importantly, though, it is prized for its high content of four main fatty acids, namely linoleic, oleic, palmitic and arachidic acid, with oleic acid being the most prominent. That being said, the fatty acid content of tiger nut oil showed 88% unsaturation. Such results show that oil extracted from tiger nuts can be a good source of edible oil and essential fatty acids[2].

Thanks to its cost- and time-efficient qualities, microwave heating is used as one of the most common procedures for food preparation today. In fact, it has been used by the food industry for drying, cooking and blanching, as well as the pasteurization and sterilization of various food products[3]. The increase in consumption of the aforementioned food products also increases the need for the use of microwave ovens for faster reheating[4]. During microwave heating, a variety of chemical changes will take place. These changes provide the lipids safety and quality indicators[5]. Oils are affected during microwave heating and, in this context, the oils degrade (oxidation, polymerization and hydrolysis)[4]. That being said, microwave-heated food products are popular due to their convenience, notably in terms of preparation time. Depending on the microwave treatment situations used, such as power, temperature and time, this heating procedure can varyingly affect lipid oxidation[6]. Subsequently, changes in amounts of minor constituents and chemical combinations affect the nutritional and functional specifications of oils. Several reports recommend that the retention of such nutrients as vitamins in microwaved foods is recovered when the heating time is shortened[6].

While there is past academic scholarship on the changes in other oils during microwave heating[3,7], there is a lack of research surrounding the effects of microwave heating on tiger nut oil.

Conducting a study in this field seems important because tiger nut oil can be a good source of edible oil and essential fatty acids. This is because tiger nut oil contains a low amount of polyunsaturated fatty acid and high amounts of oleic acid, which are sufficient for an adult's daily needs (around 10g). It also has high oxidative stability thanks to the presence of polyunsaturated fatty acids as well as gamma-tocoferol. In addition, tiger nut oil is well-recommended for cooking due to its strong resistance to chemical decomposition in high temperatures[8]. That being said, the high content of oleic and polyunsaturated fatty acids in tiger nut oil helps to decrease LDL-cholesterol (bad cholesterol) and increase HDL-cholesterol (good cholesterol) by transporting cholesterol found in the arteries to the liver for its destruction[9]. It also reduces the level of triglycerides in our blood, and it has advantageous effects on digestive secretions (biliary, pancreatic, gastric) too.

Hence, the purpose of this study is to determine whether tiger nut oil can be exploited for use as an alternative or supplement for high-quality and nutritious cooking oil in both domestic and commercial food applications. It is therefore hypothesized that high-oleic tiger nut oil possesses good thermal resistance in the cooking processes of heating and frying.

MATERIAL AND METHODS

The seeds of the brown tiger nut (*Cyperus esculentus*) plant used in this study were bought at a local market in Kano, northern Nigeria, in March. The tiger nut, with a dimension ranging from 6-10mm and coming in different varieties, is easy to find there, as it is widely distributed in climatically temperate zones, northern Nigeria being one such place. The aforementioned purchased samples from Kano were sealed in plastic bags for transport to Malaysia. The seeds were then properly sun-dried for a month before storage. Only the highest-quality seeds that were neither small nor damaged were selected and kept in a cool, dry environment at 10 °C until required. The oil of the tiger nut is solvent-extracted[10]. That being said, the chemicals and solvents used in the extraction of tiger nut oil should be analytical or HPLC-grade, obtained from BHD Laboratories (Poole, England) and Merck (Darmstadt, Germany). In addition, triacylglycerol standards (Purity 99%) were used from Sigma-Aldrich, Inc., St. Louis, California, USA. Normally, tiger nuts grown in April and picked up in November. There are mainly three varieties namely brown, yellow and black. When compared to all other varieties, the yellow variety is preferred due to its innate features such as attractive color, fleshier body, and larger size. As a result the yellow variety selected for the experiments. The heating test and the oil analysis were performed only from a well-homogenized sample (Seventy grams of oil were placed in the 100mL beaker on the microwave oven rotary turntable Tiger nut oil was heated for 2, 5, 8, 11 and 15 minutes).

Oil Extraction. Dried and cleaned tiger nuts were ground into a fine powder using Waring blender Model 32BL 80 (Dynamic Corporation of America, New Hartford, Connecticut, USA). A hundred grams of ground tiger nuts was placed into a cellulose paper cone. The oil was extracted using light petroleum ether (b.p 40-60 °C) in a 250mL soxhlet extractor for 8 hours[9]. Then, the mixture of oil and solvent was thickened with the use of rotary evaporator Model N-1 (Eyela, Tokyo Rikakikal Co., Ltd., Japan). The remaining solvent was eliminated by drying in an oven (60 °C for 1 hour) and flushing with 99.9% nitrogen. Finally, the extracted oil was kept at -20 °C prior to analysis.

Microwave heating test design. A domestic microwave oven (Sharp, Auto Cook, Malaysia) was used. Between microwave heating tests, the microwave door was opened for 5 minutes in order to cool it to ambient temperature by allowing ambient air into the microwave. This allowed the oven temperature to decrease by approximately 30 °C between tests. Seventy grams of oil were placed in the 100mL beaker on the microwave oven rotary turntable. The oil was then heated for 2, 5, 8, 11 and 15 minutes. The heated oil's temperature was determined immediately after each time period with a calibrated thermometer. The oil was later kept in storage under nitrogen at -20 °C until the commencement of analysis.

Initial oil analysis. Methods recommended by the AOCS (AOCS, 2009) were used to determine free fatty acid (FFA) (method Ca 5a-40), iodine value (method Cd 1c-85), saponification value (method Cd 3a-94), peroxide value (method Cd 8b-90) and p-anisidine value (method Cd 18-90).

PORIM test methods[11], were then used to determine viscosity, refractive index (method p4.4) and specific extinction ($E^{1\%}_{1cm}$ at 233 and 269nm) (p2.15). Total polar compounds of the oil samples were obtained according to IUPAC method 2.507 (minicolumn method)[12] and standard method (Cd 20-91). Viscosity was determined at 40 ˚C with a steel cylindrical-plate at 30 r.p.m. rotor speed. An oil sample (20mL) was poured into the sample adaptor. The attached spindle of the viscometer was then immersed into the oil. Refractive index was determined with digital refractometer Model SPER Scientific, 300034, Hong Kong, China (Brix 0-95%). Frozen tiger nut oil was first melted to a temperature of 40 ˚C and homogenized. Then, the sample was filtered by a dry Whatman No.1 filter paper to clarify the oil before the determination process. The refractive index (RI) was determined at a temperature of 40 ˚C by placing a few drops of the oil in the space between the prisms and determined values were recorded. Color was determined using the method designed by Cheikh-Rouhou *et al.*, (2007) with an Ultrascan PRO, HunterLab (Reston, USA)[13]. After that, the CieLab coordinates (L*, a*, b*) were determined with a spectrophotometer MS/Y-2500 that was calibrated with a white tile. Lightness was measured with L* value, which varies from +100 (white) to -100 (black) as well as +100 (red) to -100 (green) and +100 (yellow) to -100 (blue). Due to the increase of values a* and b*, the color of the oil became more saturated, although these values were closer to zero in terms of neutral colors (white, black or gray). That being said, a total polar compound was determined using the IUPAC method 2.507 (minicolumn method)[9].

Thermal Behavior was determined using the method of Che Man and Swe (1995) by differential scanning calorimeter using Perkins-Elmer Diamond DSC (Shelton, Connecticut, USA)[6,14]. The purge gas was nitrogen with a pressure of 20psi and flow rate of 100mL/min. The oil was first heated in an oven until completely melted. Subsequently, an estimated 5-7mg of the hot melted oil was placed in an aluminum volatile pan and hermetically sealed. The temperature of said oil was then cooled down to -60 ˚C at the rate of 5 ˚C/min and held for 2 minutes. Next, the oil was heated again from -60 to 60 ˚C at the rate of 5 ˚C/min (normal rate) and held at 60 ˚C for 2 minutes before being cooled down from 60 ˚C to -60 ˚C at the same rate (5 ˚C/min). Thermograms for heating and cooling behaviors were subsequently recorded. This was followed by the tabulation of peak, onset, offset, temperature and changes in enthalpy (ΔH) values.

Fatty acid composition was determined using the method described by the International Olive Oil Council (2001) using a gas chromatograph (Model Agilent Technologies, 6890N series, network GC system, US) equipped with a flame-lonization detector and a polar capillary column BPX70 0.25, (0.32mm internal diameter, 30m length and 0.25μm film thickness, SGE Incorporated, Austin, Texas, USA)[8]. The detector temperature was 260 ˚C, whereas the column temperature was 115 ˚C, which increased at a rate of 8 ˚C/min to 240 ˚C and was then held for 10 minutes and a run time of 36 minutes. Fatty acid composition (FAC) was determined by using the comparison of peak areas and the retention times of the standards. The peak areas were subsequently calculated and recorded in the form of percentages.

Triacylglycerol profile was determined according to the methods of AOCS CE 5b-89 (2009) using a reverse-phase high-performance liquid chromatography (HPLC) system (Perkin Elmer Series 200, Perkin Elmer, Inc., Shelton, USA) equipped with refractive index detector, pump, heater and column that are connected to a computer installed with Gilson 712 software to analyze the oil. The triacylglycerol profiles (TAG) analysis was carried out on a LiChrophase RP-C18 (5μm) column (250mm x 4mm, Merck, Darmstadt, Germany). The mobile phase used was a mixture of acetonitrile-acetone (50:50, v/v) administered at a flow rate of 1.5mL/min until a stable baseline was obtained (at least 30 minutes). The total run time was 90 minutes. The triacylglycerol profiles (TAG) were subsequently identified by a refractive index detector (220nm).

The antioxidant activity (DPPH and FRAP) was evaluated using a method designed by Botanica[15]. Following the DPPH (radical scavenging activity) method, 0.1mL of the extracted oil was mixed with 3.9mL of 80% methanol of 0.6mM DPPH. The mixture was vortexed for 20 seconds and allowed to stand for 60 minutes. The absorbance of the solutions was then measured at 515nm using a UV-Vis spectrophotometer (Genesys 10S, Thermo Scientific). The reaction was completed during this time due to no significant difference in absorption value after 60 minutes. Methanol (80%) was used as blank and a DPPH solution without any test sample (3.9mL of DPPH + 0.1mL of 80% methanol solution) was used as the control. The mean of effective concentration for radical-scavenging activity (IC_{50}) was described as mg of test sample required for a 50% decrease in absorbance of DPPH radicals. A plot of absorbance of DPPH *vs.* concentration of test sample was established to make the standard curves (dose–response curves) and to calculate IC_{50}. Based on the FRAP (Ferric Reducing Antioxidant Power) Assay, the FRAP reagent consisted of 2.5mL of 10mMolar solution of TPTZ (2,4,6- Tripyridyl-S-Triazine, Sigma) in 40mMolar HCl plus 2.5mL of 20mMolar $FeCl_3$ and 25mL of 0.3mMolar acetate buffer (pH 3.6). The reagent was freshly prepared and kept at 37°C. Then, 40μL of sample extract was mixed with 0.2mL purified water and 1.8mL of FRAP reagent. After 10 minutes' incubation at 37°C, the absorbance of mixture was spectrophotometrically determined at 593nm. The calibration curve of $FeSO_4$ solution was drawn as a standard solution. The result was described as the concentration of an antioxidant having a ferric reducing capacity equivalent to that of 1mMolar solution of $FeSO_4$.

Statistical analysis. All values presented are means of triplicate determination with standard deviations. Statistical analyses were performed using Minitab 16 software and ANOVA. Significant differences between values are at $p<0.05$ levels.

RESULTS

The temperatures of tiger nut oil during microwave heating were 25 °C (laboratory temperature), 92 °C, 202 °C, 212 °C, 225 °C and 240 °C after 0, 2, 5, 8, 11 and 15 minutes, respectively. Changes in viscosity with time are presented in Table 1.

The effect of microwave heating on thermal behavior of tiger nut oil is shown in Table 2. In both heating and cooling curves, there were 2 peaks.

Table 1. Changes in viscosity and refractive index in tiger nut oil during microwave heating.

Time (min)	Viscosity (Cp)	Refractive index (n_D40 °C)	Color		
			L*	a*	b*
0	32.73 ± 0.03^{f}	1.4650 ± 0.00^{c}	52.7 ± 0.28^{a}	-3.73 ± 0.02^{a}	50.07 ± 0.21^{a}
2	36.53 ± 0.04^{e}	1.4661 ± 0.00^{b}	48.02 ± 0.45^{b}	-3.93 ± 0.03^{b}	39.09 ± 0.85^{b}
5	39.30 ± 0.14^{d}	$1.4668\pm0.00^{a,b}$	46.05 ± 1.01^{c}	-4.47 ± 0.04^{c}	28.16 ± 0.15^{c}
8	41.21 ± 0.02^{c}	1.4669 ± 0.00^{a}	42.45 ± 0.24^{d}	-4.84 ± 0.03^{d}	21.05 ± 0.03^{d}
11	44.71 ± 0.04^{b}	1.4670 ± 0.00^{a}	33.95 ± 0.24^{e}	-5.13 ± 0.01^{e}	14.87 ± 0.46^{e}
15	46.46 ± 0.08^{a}	1.4674 ± 0.00^{a}	28.15 ± 0.05^{f}	-5.25 ± 0.04^{e}	9.12 ± 0.04^{f}

M±SD= Mean ± standard deviation of triplicate readings.
Results in column followed by same letter superscript are not significantly different.

Table 2. DSC melting and crystallization temperatures of tiger nut oil during microwave heating.

		Melting				Crystallization			
		Onset (°C)	Peak (°C)	End set (°C)	Peak area (mJ)	Onset (°C)	Peak (°C)	End set (°C)	Peak area (mJ)
0 oil	**Peak 1**	-9.01	-0.24	5.47	829.67	-3.81	-6.22	-12.99	-47.34
	Peak 2	8.74	11.88	13.97	58.66	-40.82	-44.19	-47.72	-340.19
2 min	**Peak 1**	-7.90	-0.41	5.38	783.80	-3.61	-4.25	-10.34	-33.55
	Peak 2	9.84	12.13	14.00	36.28	-38.52	-41.86	-45.60	-329.18
5 min	**Peak 1**	-6.10	0.59	4.61	646.36	-2.77	-4.00	-10.67	-41.16
	Peak 2	9.55	12.04	13.89	40.41	-40.00	-43.69	-47.38	-279.55
8 min	**Peak 1**	-6.04	1.40	5.57	611.70	-2.50	-4.31	-9.75	-29.50
	Peak 2	10.20	12.78	14.55	34.70	-40.10	-43.92	-47.82	-258.66
11 min	**Peak 1**	-5.58	1.43	5.16	637.15	-2.19	-4.48	-11.80	-55.49
	Peak 2	10.45	12.73	14.40	29.25	-39.90	-44.35	-48.85	-281.28
15 min	**Peak 1**	-5.74	-0.43	2.35	473.97	-1.72	-3.29	-9.79	-40.87
	Peak 2	4.57	6.96	9.32	5.54	-45.22	-50.39	-55.86	-155.96

Table 3 illustrates the effect of free fatty acid (FFA) of tiger nut oil during microwave heating. Iodine value is the measure of the unsaturation of fats and oils. Iodine values of tiger nut oil are shown in Table 3. Changes in peroxide value during microwave heating are shown in Table 3. Changes in the total oxidation value (TOTOX) of tiger nut oil during microwave heating are shown in Table 3. Table 3 illustrates the effect of microwave heating on total polar compounds (TPC). Table 3 illustrates the changes in specific extinction ($E_{1cm}^{1\%}$) at 233 and 269nm during the microwave heating of tiger nut oil. The specific extinction at 269nm did not actually change too much during microwave heating. Here, the UV spectrophotometric analysis represents the grade of tiger nut oil oxidation.

Table 4 shows the effects of microwave heating on fatty acid compositions (FAC).

Table 5 shows small but significant modifications observed regarding the decline in the triacylglycerol profiles (TAG) percentage.

The results of antioxidant activity by DPPH test are shown in Table 6.

Table 7 illustrates the ferric reducing activity of tiger nut oil during microwave heating.

Table 3. Changes in FFA, IV, SV, PV, p-AV, TOTOX, TPC and specific extinction in tiger nut oil during microwave heating.

Time (min)	FFA (%)	IV (g I_2/100 g oil)	SV (mg KOH/g)	PV	p-AV	TOTOX	TPC (%)	Specific Extinction	
								$E_{1\,cm}^{1\,\%}$ at 233 nm	$E_{1\,cm}^{1\,\%}$ at 269 nm
0	0.10 ± 0.00^{c}	74.90 ± 0.09^{a}	192.81 ± 0.02^{d}	3.06 ± 0.05^{e}	0.72 ± 0.04^{f}	6.84 ± 0.16^{e}	3.11 ± 0.20^{d}	2.68 ± 0.00^{f}	0.27 ± 0.00^{f}
2	0.11 ± 0.00^{b}	73.48 ± 0.10^{b}	192.89 ± 0.03^{c}	4.02 ± 0.01^{d}	1.54 ± 0.03^{e}	9.58 ± 0.06^{d}	$3.48 \pm 0.23^{c,d}$	3.07 ± 0.00^{e}	0.34 ± 0.00^{e}
5	0.11 ± 0.00^{b}	72.62 ± 0.04^{c}	$192.96 \pm 0.00^{b,c}$	5.43 ± 0.01^{b}	3.10 ± 0.28^{d}	13.96 ± 0.31^{c}	$4.35 \pm 0.21^{b,c}$	3.22 ± 0.04^{d}	0.56 ± 0.00^{d}
8	0.11 ± 0.00^{b}	72.09 ± 0.09^{d}	192.95 ± 0.00^{c}	5.84 ± 0.02^{a}	5.63 ± 0.02^{c}	17.31 ± 0.08^{b}	4.84 ± 0.12^{b}	3.41 ± 0.00^{c}	0.85 ± 0.00^{c}
11	0.12 ± 0.00^{a}	71.03 ± 0.02^{e}	$193.03 \pm 0.02^{a,b}$	5.21 ± 0.01^{c}	7.44 ± 0.16^{b}	$17.86 \pm 0.19^{a,b}$	6.05 ± 0.24^{a}	3.84 ± 0.00^{b}	1.01 ± 0.01^{b}
15	0.12 ± 0.00^{a}	69.69 ± 0.11^{f}	193.09 ± 0.00^{a}	4.11 ± 0.00^{d}	10.02 ± 0.02^{a}	18.25 ± 0.03^{a}	6.74 ± 0.53^{a}	4.36 ± 0.02^{a}	1.24 ± 0.00^{a}

M±SD= Mean ± standard deviation of triplicate readings.
Results in column followed by same superscript letter are not significantly different.

Table 4. Fatty acid composition of tiger nut oil during microwave heating.

Time (min)	Fatty acid composition (%)								
	$C_{14:0}$	$C_{16:0}$	$C_{16:1}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{20:0}$	$C_{22:0}$	$C_{24:0}$
0	0.12 ± 0.00^{a}	17.04 ± 0.04^{e}	0.31 ± 0.00^{a}	8.74 ± 0.01^{e}	61.02 ± 0.02^{a}	10.91 ± 0.06^{a}	1.51 ± 0.00^{c}	0.13 ± 0.00^{d}	0.20 ± 0.00^{c}
2	0.12 ± 0.00^{a}	17.69 ± 0.19^{d}	$0.30 \pm 0.00^{a,b}$	9.20 ± 0.07^{d}	60.32 ± 0.13^{b}	$10.48 \pm 0.00^{a,b}$	1.52 ± 0.00^{c}	$0.14 \pm 0.00^{c,d}$	$0.20 \pm 0.00^{b,c}$
5	0.12 ± 0.00^{a}	18.12 ± 0.02^{c}	$0.30 \pm 0.00^{a,b,c}$	9.48 ± 0.02^{c}	59.81 ± 0.22^{c}	10.26 ± 0.13^{b}	1.53 ± 0.02^{c}	0.14 ± 0.00^{c}	$0.21 \pm 0.00^{b,c}$
8	0.12 ± 0.00^{a}	18.30 ± 0.02^{c}	$0.29 \pm 0.00^{b,c,d}$	9.63 ± 0.03^{c}	59.52 ± 0.05^{c}	$10.12 \pm 0.07^{b,c}$	1.61 ± 0.02^{b}	0.17 ± 0.00^{b}	$0.22 \pm 0.00^{a,b,c}$
11	0.12 ± 0.00^{a}	18.97 ± 0.12^{b}	$0.28 \pm 0.00^{c,d}$	9.96 ± 0.12^{b}	58.84 ± 0.01^{d}	$9.69 \pm 0.02^{c,d}$	1.71 ± 0.01^{a}	$0.18 \pm 0.00^{a,b}$	$0.23 \pm 0.00^{a,b}$
15	0.13 ± 0.00^{a}	19.40 ± 0.04^{a}	0.28 ± 0.00^{d}	10.30 ± 0.04^{a}	58.27 ± 0.09^{e}	9.42 ± 0.01^{d}	1.75 ± 0.00^{a}	0.19 ± 0.00^{a}	0.24 ± 0.00^{a}

$\mathbf{C_{14:0}}$ = Myristic acid; $\mathbf{C_{16:0}}$ = Palmitic acid; $\mathbf{C_{16:1}}$ = Palmitoleic acid; $\mathbf{C_{18:0}}$ = Stearic acid; $\mathbf{C_{18:1}}$ = Oleic acid; $\mathbf{C_{18:2}}$ = Linoleic acid; $\mathbf{C_{20:0}}$ = Arachidic acid; $\mathbf{C_{22:0}}$ = Behinic acid; $\mathbf{C_{24:0}}$ = Lignoceric acid.

M±SD = Mean ± standard deviation of triplicate readings.
Results in column followed by same superscript letter are not significantly different.

Table 5. Triacylglycerol composition (TAG) of tiger nut oil during microwave heating.

TAG specie	**TAG composition (%)**					
	0 min	**2 min**	**5 min**	**8 min**	**11 min**	**15 min**
LLL	0.09±0.00^{a}	0.09±0.00^{a}	0.09±0.00^{a}	0.09±0.00^{a}	0.08±0.00a	0.07±0.00^{a}
OLLn+PoLL	0.03±0.00^{e}	0.03±0.00^{d}	0.03±0.00^{c}	0.03±0.00^{b}	0.03±0.00^{a}	0.02±0.00^{f}
PLLn	0.04±0.00^{d}	0.04±0.00^{c}	0.04±0.00^{b}	0.03±0.00^{f}	0.04±0.00^{a}	0.03±0.00^{e}
OLL+OLLn	1.49±0.00^{b}	1.47±0.00^{b}	1.42±0.00^{c}	1.38±0.00^{d}	1.32±0.00^{f}	1.81±0.00^{a}
PLL	0.52±0.00^{a}	0.51±0.00^{a,b}	0.5±0.00^{b,c}	0.51±0.00^{b,c}	0.49±0.00^{c}	0.47±0.00^{d}
POLn	0.22±0.00^{d}	0.27±0.00^{a,b}	0.23±0.00^{c,d}	0.22±0.00^{d}	0.28±0.00^{a}	0.25±0.00^{b,c}
LOO+PLnP	8.85±0.00^{a}	8.77±0.00^{a}	8.7±0.00^{b}	8.61±0.00^{c}	8.54±0.01^{d}	8.49±0.02^{e}
PoOO+PLO+SLL+PoOP	6.14±0.01^{a}	6.07±0.01^{b}	5.99±0.01^{c}	5.71±0.00^{e}	5.77±0.00^{d}	5.65±0.00^{f}
PLP	0.59±0.00^{c}	0.59±0.00^{c}	0.65±0.00^{b}	0.74±0.00^{a}	0.52±0.00^{d}	0.65±0.00^{b}
OOO	33.75±0.00^{a}	33.23±0.01^{b}	32.95±0.01^{c}	32.85±0.00^{d}	32.66±0.01^{e}	31.67±0.02^{f}
POO	25.28±0.00^{a}	25.05±0.02^{b}	24.98±0.01^{b}	24.78±0.03^{c}	24.32±0.02^{d}	23.93±0.02^{e}
POP	3.6±0.02^{e}	3.88±0.01^{d}	3.91±0.01^{d}	4.04±0.02^{c}	4.39±0.02^{b}	4.94±0.02^{a}
OOG	0.26±0.00^{a}	0.25±0.00^{a}	0.21±0.00^{b}	0.23±0.00^{b}	0.15±0.00^{c}	0.12±0.00^{d}
SOO	11.78±0.01^{a}	11.54±0.02^{b}	11.47±0.01^{c}	11.43±0.01^{c}	11.22±0.01^{d}	11.11±0.01^{e}
POS	3.34±0.00^{e}	3.33±0.00^{e}	3.51±0.00^{d}	3.67±0.01^{c}	4.37±0.00^{b}	4.63±0.01^{a}
OOA	1.94±0.01^{a}	1.93±0.00^{a,b}	1.89±0.00^{b,c}	1.87±0.00^{c}	1.75±0.01^{d}	1.63±0.00^{e}
SSO+POA	2.08±0.01^{f}	2.95±0.02^{e}	3.43±0.01^{d}	3.81±0.02^{c}	4.07±0.02^{b}	4.53±0.01^{a}

A = arachidic; **G =** gadolic; **L=** linoleic; **Ln =** linolenic; **O =** oleic; **P =** palmitic; **Po =** palmitoleic; **S =** stearic.

Table 6. Changes in the antioxidant activity of tiger nut oil during microwave heating assessed by the DPPH assay (inhibition %).

Concentration (ppm)	**0 min**	**2 min**	**5 min**	**8 min**	**11 min**	**15 min**
5000	68.60±0.93^{a}	63.53±0.79^{a}	48.09±0.70^{a}	37.83±0.83^{a}	33.17±0.94^{a}	19.66±0.77^{a}
2500	41.86±1.39^{b}	39.35±0.98^{b}	35.55±0.69^{b}	25.86±1.08^{b}	24.85±0.68^{b}	10.98±0.67^{b}
1250	31.25±1.01^{c}	27.09±0.86^{c}	26.66±0.93^{c}	19.29±0.49^{c}	16.37±0.89^{c}	4.03±0.52^{c}
625	23.51±0.97^{d}	20.00±0.56^{d}	16.20±0.89^{d}	12.39±0.73^{d}	8.02±0.65^{d}	ND
312.5	16.27±0.75^{e}	14.40±1.19^{e}	12.11±0.44^{e}	7.31±0.77^{e}	6.52±1.58^{e}	ND
156.25	11.68±1.73^{f}	10.39±0.86^{f}	8.38±0.86^{f}	5.23±0.65^{f}	1.00±0.65^{f}	ND
78.13	9.89±1.93^{g}	7.74±1.29^{g}	4.65±0.97^{g}	2.93±1.31^{g}	ND	ND
IC_{50}	**3242.39**	**3608.97**	**4760.79**	**6409.11**	**7143.71**	**12046.24**

M±SD = Mean ± standard deviation of triplicate readings.
Results in column followed by same superscript letter are not significantly different.

Table 7. Changes in the antioxidant activity of tiger nut oil during microwave heating assessed by the FRAP assay (μM($FeSO_4 \cdot 7H_2O$)/g sample).

Time (min)	μM($FeSO_4 \cdot 7H_2O$)/g sample
0	105.81±0.09^a
2	102.05±0.07^b
5	95.13±0.07^c
8	94.57±0.07^d
11	91.78±0.09^e
15	83.72±0.10^f

M±SD = Mean ± standard deviation of triplicate readings.
Results followed by same superscript letter are not significantly different.

DISCUSSION

Temperature of the oil during microwave heating

Certainly, the high temperature achieved affects the oil's physic-chemical properties.

Changes in viscosity, refractive index and color during microwave heating

Viscosity increase is a temperature-dependent factor justified by chemical reactions that occur within the oils. The cyclic monomers, dimmers and polymers formation in a non-radical mechanism leads to the increase of viscosity. The formation of conjugated dienes happens when the double bonds are transferred within the unsaturated fatty acids. These impermanent constructions are responsible for the formation of ring constructions, permitting bonds among two triacylglycerols and therefore resulting in the formation of bigger molecules. Here, the ratio of saturated fatty acids (which adds further viscosity due to their higher melting point) increases as unsaturated fatty acids undergo oxidation. That being said, the oxygen interpolation in the fatty acids (oxidized triglycerides) corresponds to the increased density[5].

This finding is in line with Albi y Col.[16] who posited that, in contrast to usual liquids, a rise in viscosity is considerable when edible fats and oils are heated. Here, they revealed that, as opposed to conventional heating, microwave heating causes a higher increase in viscosity. In this context, the results obtained from the oils subjected to microwave energy without any rise in temperature, were totally identical to those labeled as unheated oils[3].

Refractive index increased gradually during microwave heating and the increases were significant ($p<0.05$). The refractive index was within the range reported for vegetable oils: rapeseed oil (1.465–1.469), pumpkin seed oil (1.466–1.474), olive oil (1.468–1.471) and sunflower seed oil (1.467–1.469)[17]. The refractive index (RI) is often used as a criterion for purity. At a temperature above 40°C, the refractive index of unsaturated fatty acid increases with the degree of unsaturation, while the refractive index of saturated fatty acid shows a linear increase with escalating chain length. This has been proven in another past study by Khan y Cols., in which they detected a small increase in refractive index of some edible oils (such as corn, soybean, coconut and mustard) after microwave heating[18].

Owing to the high amounts of carotenoids and pigments that were extracted into the oil, the tiger nut oil has a dark color (Table 1). Furthermore, there was a significant ($p<0.05$) decrease in L*, a* and b* parameters during heating. During the first 3 minutes of microwave heating, color parameters were quite resistant, but began to show signs of breaking down after 5 minutes, with the exclusion of transparency wherein a slow elevation was considered with the increase in duration of exposure. That being said, the color of tiger nut oil during 8 minutes of microwave heating changed slowly from yellow to brown. The transition to dark brown was eventually observed at 11-15 minutes. Here, the darkening of seed oils was due to the presence of phospholipids. In this context, seed oils darken during the heating trial, and the rate of darkening is proportional to the heating time. At this point, it ought to be mentioned that a burnt odor was detected after 5 minutes of heating. Conversely, though, one of the important sensorial features of oil that is observed by the consumer is color, as it plays an integral role in its commercial viability. These findings are in line with Abdulkarim y Cols., who posited that changes in color among frying oils are a visual indication of the extent in which such oils have experienced deterioration due to oxidation[14].

Melting and crystallization behavior

Given that the melting point of oils is related to their physical properties like thermal behavior and hardness, the changes in the peak sizes of peak 2 indicate that the high-melting triacylglycerol profiles (TAGs) are reduced or converted to a lower melting type. Generally, at higher temperatures, the trends would become more pronounced. Thermal features might thus be correlated to the formation of primary lipid oxidation products only for those fats and oils that exhibit wide lipid oxidation after microwave treatment.

All cooling curves for tiger nut oil during microwave heating have two distinct exothermic peaks that may correspond to 2 triacylglycerol profile (TAG) categories (mono and diunsaturated TAGs).

The attendance of two separate peaks may be due to the presence of a second polymorphic form. The oils were exposed to microwave heating, and such products of oxidation as polar compounds, aldehydes, hydroperoxydes, dimers and polymers might disestablish the rearrangement of various polymorphic forms in oils[19]. It is therefore necessary to know the chemical composition, physical properties and the thermal behavior of edible oils for setting up standards within each specific usage as well as an accurate control of processes[20]. Differential scanning calorimetry (DSC) can be consumed to explain the thermal behavior of edible oils.

Changes in free fatty acid (FFA)

Undoubtedly, the increase in free fatty acid (FFA) was due to the breaking of the ester linkages of triglyceride molecules as a consequence of heating[21]. These findings are in line with all results that show how microwave heating causes little increase in the amount of free fatty acids (FFA) and it was more than the results obtained through conventional heating. In fact, in a past study by Cerretani y Cols., where extra virgin olive oil was compared with other types of olive oil, it was found that the free fatty acid (FFA) content of extra virgin olive oil increased after 12 minutes of microwave heating, although this occurred at acutely high temperatures (over 300 °C)[8].

Changes in iodine value (IV) and saponification value (SV)

The reduction of iodine values through microwave treatment may be ascribable to reductions in the amount of unsaturation sites as a result of oxidation, polymerization or breakage of long-chain fatty acids[17]. These findings are in line with Abdulkarim y Cols.[14], who posited that a decrease in iodine levels can be attributed to the disintegration of double bounds by oxidation, scission and polymerization.

Saponification values (SV) of oil have been known to increase significantly ($p<0.05$) during microwave treatment. The increase in the saponification value of oils may result from the increase in free fatty acids in the seeds as a result of triglyceride breakdown. Also, high temperature is known to result in remarkable free fatty acid formation from triglycerides. Thus, the saponification values of crude tiger nut oil are within the standards set for refined oil (185-196)[12]. The high amount of saponification value in crude tiger nut oil is evident based on its tendency to foam when subjected to heat, and this makes it a suitable ingredient for soaps as well.

Changes in peroxide value (PV), p-anisidine value (p-AV) and the total oxidation value (TOTOX)

Past academic scholarship has shown that vegetable oils with a peroxide value (PV) of 7.5meq/kg tend to have unacceptable sensory attributes[5]. Microwave heating has a significant ($p<0.05$) effect on peroxide value (PV) that can be explained by changes during the oxidation process, which may potentially reach the maximum point due to hydroperoxide formation and then decline when the formations of oxidation's byproducts occur. Hence, the peroxide value (PV) was selected as a unit of measurement for oxidation levels during the stage of microwave heating. These findings are in line with Yoshida y Cols.[21] that impermanent peroxides decompose quickly to secondary oxidation products. During lipid oxidation, the primary reaction products (hydroperoxides) were decomposed to produce secondary oxidation products such as ketones, alcohols, acids, aliphatic and hydrocarbons that are more permanent during the heating procedure[11].

As it is shown in Table 3, the p-anisidine value (p-AV) increased significantly ($p<0.05$) during microwave heating from its initial value of 0.72 reaching the highest value of 10.2 after 15 minutes. These findings are in line with Abdulkarim y Cols.[14], who posited that the oxidative deterioration of oil and its initial fatty acid composition will affect p-anisidine value (p-AV).

The increase in the total oxidation value (TOTOX) during microwave heating provides information with regards to developments in the formation of primary and secondary oxidation products. According to the results, the formation of primary and secondary lipid oxidation products has occurred.

These findings are in line with Poiana[11], who posited that the total oxidation value (TOTOX) for oil samples exposed to convection and microwave heating treatments, with increasing heating time, showed a marked increase. This also confirms the compatibility of the total oxidation value (TOTOX) as the mathematical prediction of oxidative stability, thereby deeming it suitable as a sign of overall oxidative stability that is also correlated with the extent of all deterioration[22].

Fatty acid composition (FAC)

Effects of microwave heating on fatty acid compositions (FAC) are characteristic of vegetable oils, such as linoleate, linolenate and methyl oleate. Specifically, the microwave heating of tiger nut oil resulted in a significant ($p<0.05$) decrease in $C_{16:1}$, $C_{18:1}$ and $C_{18:2}$ and after them ratios during microwave oven heating. The decrease is due to a decline in polyunsaturated fatty acids by oxidation. This parameter was considered to be a reliable indicator of lipid oxidation during the heating process. Similar patterns were observed by Tan y Col.[23], and Abdulkarim[14]. Caponio y Cols.[7] had also evaluated the fatty acid composotion (FAC) of 3 vegetable oils (peanut oil, virgin olive oil and sunflower oil) and, after both microwave heating and conventional heating, found that the saturated fatty acids did not change significantly after heating, while unsaturated fatty acids and polyunsaturated fatty acids were considerably reduced.

Triacylglycerol profile

Until now there have been no studies on how microwave heating effects on the fatty acid distribution of triacylglycerols (TAGs). Yeboah *et al.*, (2012), however, have reported 7 major triacylglycerol profiles (TAG) classes in tiger nut oil, with $C_{54:3}$ (29.00%) and $C_{52:2}$ (27.82%) being the dominant ones[24]. It has been shown that the oils are mixtures of triesters of glycerol (triacylglycerols or TAGs) with a high amount of double and triple unsaturated fatty acids[25].

Total polar compounds (TPC) are represented by all the substances that are higher in polarity with respect to that of the unchanged triglycerides. The increase of polar compounds in the thermally-treated oils are largely attributed to the triglyceride oligopolymers and oxidised triglycerides, which significantly increased ($p<0.001$) with heating time during microwave treatment. These findings are in line with Abdulkarim y Cols.[14], who posited that total polar compounds (TPC) tend to increase with heating time. In contrast with triacylglycerol profiles (TAG), total polar compounds (TPC) demonstrate all the products of fatty acid degradation with greater polarity and contain oxidized and polymerized triacylglycerol profiles (TAG), in addition to hydrolysis products as free fatty acids and diglycerides[20].

Changes in specific extinction ($E^{1\%}_{1cm}$ at 233 and 269nm)

Changes in the ultraviolet absorption at 233 and 269nm are related to changes in the amount of conjugated dienes and conjugated trienes, respectively. These were present in the oil due to the oxidation of polyunsaturated fatty acids. The results of conjugated dienes and conjugated trienes represent an intense absorption at 233 and 269nm respectively. Secondary oxidation compounds were not detected before reaching the oxidation induction time. This confirmed that tiger nut oil has a high resistance against heating. The primary oxidation products are unstable under heating and their degradation could promote the formation of secondary oxidation products, which have an absorption rate at about 269nm[19].

Radical scavenging effect (DPPH)

Using DPPH reagent, the free radical scavenging capability of the antioxidant can be evaluated by spectrophotometric methods[10].

Generally, reactions of oxidation in organic systems would create free radicals that start off chain reactions. Antioxidants terminate these chain reactions perhaps when they are oxidized themselves or by eliminating free radical intermediates.

At the beginning of the heating process, free radicals are formed in small amounts. Nevertheless, these free radicals are immediately neutralized by antioxidants that are present in the oil, but antioxidants are slowly consumed during heating. As heat travels from areas with low concentration of antioxidants to those with higher concentration, the induction period increases. However, it becomes lower in areas with high concentrations of antioxidants. The antioxidant activity in areas where it is most concentrated may reduce in some cases and, as a result, the antioxidant becomes a prooxidant. Hence, it is not reasonable to add antioxidants in high amounts[9].

Ferric reducing antioxidant power (FRAP)

The presence of reducers (antioxidants) causes the reduction of Fe3+/ferricyanide complex to its ferrous form (Fe2+). The most significant point of reduction was detected when the oil was exposed to microwave heating after 15 minutes. That being said, the analysis of antioxidant activity was accomplished using the FRAP (ferric-reducing antioxidant power) method described. The results are expressed as ascorbic acid equivalent antioxidant capacity. The reducing power of tiger nut oil is a dependable index of antioxidant activity, showing that the antioxidant compounds are electron donors and can decrease the oxidized intermediates of lipid peroxidation[11].

CONCLUSIONS

High-oleic tiger nut oil has demonstrably higher values in experiments that measure oxidative stability. It is comparable with most other oils used in domestic and commercial applications with the added advantage of containing high amounts of monounsaturated fatty acids (oleic acid) that has been credited with preventing the risks of both heart disease and high cholesterol. Tiger nut oil can therefore be exploited for use as an alternative or supplementary source of high-quality and nutritious cooking oil for both domestic and commercial culinary purposes.

CONFLICTO DE INTERESES

Authors state that there are no conflicts of interest in preparing the manuscript.

REFERENCES

(1) Ezeh O. Edible oil from Tiger nut (Cyperus esculentus L.): mechanical pressing and aqueous enzymatic extraction methods [PhD thesis]. [Reading, United Kingdom]: University of Reading; 2016. Disponible en: http://centaur.reading.ac.uk/59583/

(2) Muhammad NO, Bamishaiye EI, Usman LA, Salawu MO, Nafiu MO, Oloyede OB. Physicochemical properties and fatty acid composition of cyperus esculentus (Tiger Nut) Tuber Oil. Biores Bull. 2011; 5: 51-4.

(3) Albi T, Lanzón A, Guinda A, Pérez-Camino MC, León M. Microwave and Conventional Heating Effects on Some Physical and Chemical Parameters of Edible Fats. J Agric Food Chem. 1997; 45(8): 3000-3.

(4) Inchingolo R, Cardenia V, Rodriguez-Estrada MT. The effects of microwave heating on edible oils and lipid-containing food. Lipid Technol. 2013; 25(3): 59-61.

(5) Malheiro R, Casal S, Ramalhosa E, Pereira JA. Chapter 26: Microwave Heating: A Time Saving Technology or a Way to Induce Vegetable Oils Oxidation? En: Grundas S, editor. Advances in Induction and Microwave Heating of Mineral and Organic Materials. London: InTech; 2011. p. 597-614. Disponible en: http://www.intechopen.com/books/advances-in-induction-and-microwave-heating-of-mineral-and-organic-materials/microwave-heating-a-time-saving-technology-or-a-way-to-induce-vegetable-oils-oxidation-

(6) Anjum F, Anwar F, Jamil A, Iqbal M. Microwave roasting effects on the physico-chemical composition and oxidative stability of sunflower seed oil. J Amer Oil Chem Soc. 2006; 83(9): 777-84.

(7) Caponio F, Pasqualone A, Gomes T. Changes in the fatty acid composition of vegetable oils in model doughs submitted to conventional or microwave heating. Int J Food Sci Technol. 2003; 38(4): 481-6.

(8) Cerretani L, Bendini A, Rodriguez-Estrada MT, Vittadini E, Chiavaro E. Microwave heating of different commercial categories of olive oil: Part I. Effect on chemical oxidative stability indices and phenolic compounds. Food Chem. 2009; 115(4): 1381-8.

(9) Akoh CC, Lai O-M. Healthful lipids. Champaign, Ill.: AOCS Press; 2005.

(10) Akoh CC, Min DB. Food lipids: chemistry, nutrition, and biotechnology. 3rd ed. Boca Raton, FL: CRC Press/Taylor & Francis Group; 2008.

(11) Poiana M-A. Enhancing oxidative stability of sunflower oil during convective and microwave heating using grape seed extract. Int J Mol Sci. 2012; 13(7): 9240-59.

(12) Oomah BD, Liang J, Godfrey D, Mazza G. Microwave Heating of Grapeseed: Effect on Oil Quality. J Agric Food Chem. 1998; 46(10): 4017-21.

(13) Cheikh-Rouhou S, Besbes S, Hentati B, Blecker C, Deroanne C, Attia H. Nigella sativa L.: Chemical composition and physicochemical characteristics of lipid fraction. Food Chem. 2007; 101(2): 673-81.

(14) Abdulkarim SM, Long K, Lai OM, Muhammad SKS, Ghazali HM. Frying quality and stability of high-oleic Moringa oleifera seed oil in comparison with other vegetable oils. Food Chem. 2007; 105(4): 1382-9.

(15) Buonincontri M, Allevato E, di Pasquale G. The problem of the alternating dominance of deciduous and evergreen vegetation: archaeo-anthracological data from Northern Maremma. Ann Bot. 2013; 3: 165-71.

(16) Albi T, Lanzón A, Guinda A, Pérez-Camino MC, León M. Microwave and Conventional Heating Effects on Some Physical and Chemical Parameters of Edible Fats. J Agric Food Chem. 1997; 45(8): 3000-3.

(17) Uquiche E, Jeréz M, Ortíz J. Effect of pretreatment with microwaves on mechanical extraction yield and quality of vegetable oil from Chilean hazelnuts (Gevuina avellana Mol). Innov Food Sci Emerg Technol. 2008; 9(4): 495-500.

(18) Khan MN, Sarwar A, Wahab MF. Chemometric assessment of thermal oxidation of some edible oils. J Therm Anal Calorim. 2010; 102(1): 369-74.

(19) Besbes S, Blecker C, Deroanne C, Lognay G, Drira N-E, Attia H. Heating effects on some quality characteristics of date seed oil. Food Chem. 2005; 91(3): 469-76.

(20) Santos JCO, Santos MGO, Dantas JP, Conceição MM, Athaide-Filho PF, Souza AG. Comparative study of specific heat capacities of some vegetable oils obtained by DSC and microwave oven. J Therm Anal Calorim. 2005; 79(2): 283-7.

(21) Yoshida H, Tatsumi M, Kajimoto G. Influence of fatty acids on the tocopherol stability in vegetable oils during microwave heating. J Am Oil Chem Soc. 1992; 69(2): 119-25.

(22) Pereira de Abreu DA, Losada PP, Maroto J, Cruz JM. Evaluation of the effectiveness of a new active packaging film containing natural antioxidants (from barley husks) that retard lipid damage in frozen Atlantic salmon (Salmo salar L.). Food Res Int. 2010; 43(5): 1277-82.

(23) Tan CP, Man YBC. Differential scanning calorimetric analysis of edible oils: Comparison of thermal properties and chemical composition. J Amer Oil Chem Soc. 2000; 77(2): 143-55.

(24) Yeboah SO, Mitei YC, Ngila JC, Wessjohann L, Schmidt J. Compositional and structural studies of the oils from two edible seeds: Tiger nut, Cyperus esculentum, and asiato, Pachira insignis, from Ghana. Food Res Int. 2012; 47(2): 259-66.

(25) van den Berg JDJ, Vermist ND, Carlyle L, Holčapek M, Boon JJ. Effects of traditional processing methods of linseed oil on the composition of its triacylglycerols. J Sep Science. 2004; 27(3): 181-99.