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EFFECT OF FRYING AND MODE OF STORAGE ON THE QUALITY OF SESAME (*Sesamun indicum* L.) OIL

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ABSTRACT
The present study investigated the effect of frying and mode of storage on the quality of sesame oil. This study is of great relevance as it reveals the effect of storage at room temperature and refrigeration on the valuable properties of the sesame oil (fried and un-fried) such as; physicochemical properties, antioxidant properties, and pigment composition. Sesame seed oil was extracted using cold press method. A portion of the extracted oil was fried at 100 ^{o}C for 60 min. The aliquots of fried and the un-fried oils were separately dispensed into clean transparent bottles of equal size/volume and twenty (20) bottles each of the fried and un-fried oils were stored both at an ambient (30 ^{o}C) and refrigerated temperature (-4 ^{o}C) for 16 days. The oil samples were analyzed for acid value, saponification value and density at 4 days interval. Result shows that frying and storage at ambient temperature decreased the density and specific gravity of the sesame oil. However, frying increased their acid value (AV) and saponification value (SV) of sesame oil stored at ambient and refrigerated temperature with slight change in AV when the storage condition was varied on the 16th day. A relative increase in the SV was also observed as the fried oil aged from 8 to 16 days at ambient temperature. The carotenoid content of oils stored in refrigerator were higher than oils stored at ambient temperature. Frying decreased the carotenoids content of the sesame oils. The fried sesame oil had higher radical scavenging activity than the un fried oil. This indicates that fried sesame oils may have longer shelf life due to its increased radical scavenging activities. However, the radical scavenging activity of the fried sesame oil stored at ambient temperature was higher than that stored at refrigeration temperature. It is concluded that frying decreased the carotenoid content and antioxidant activity of sesame seed oil. Refrigeration increased the physicochemical properties but decreased the antioxidant activity of sesame seed oil.

Keywords: *Sesame oil, mode of storage, Frying, ambient temperature, refrigerated temperature, acid value, saponification values, DPPH radical scavenging, specific gravity, density, carotenoids.*

INTRODUCTION
Sesame *(Sesamum indicum*L.), commonly known as beniseed, is a tropical herbaceous annual plant belonging to the family *Pedaliaceae*. Sesame is generally believed to have originated from Africa, probably West Africa, from where it spreads through Western Asia to India, China, and Japan and to other parts of the world (Nayar and Mehra, 1970; Bedigian, 2003). In Nigeria, sesame is widely spread, borne out by the fact that there are over twenty different names in different languages for the crop. It is called Ridi (Hausa), Isasa (Igbo), Ekuku or Eeku (Yoruba), Ocha (Idoma), Igogo (Igala) etc. Its colour varies from cream-white to charcoal-black but it is mainly white or black. The physical properties of sesame seeds vary probably as a result of variability in genotypic effects (El Khier*et al.*, 2008). The physical properties i.e length, width, thickness, geometric mean diameter, sphericity and surface area of local sesame seeds varieties in Nigeria varied from 2.9-3.2, 1.9-2.1, 0.85-0.91, 1.59-1.72, 0.575-0.58 and 7.05-10.2mm^2 respectively (Tunde-Akintunde and Akintunde, 2007). The sesame plant and seeds are shown in Figure 1 below. Main areas of cultivation in Nigeria include Jigawa, Benue, Kwara, Kogi, Nasarawa and Niger States. Sesame is an important export crop in Nigeria, and Nigeria has a substantial role in the global beniseed trade (Tunde-Akintunde *et al.*, 2012).

Sesame contains many health benefiting nutrients, minerals, antioxidants and vitamins (Borchani *et al.*, 2010). Sesame is grown for its seeds which are excellent source of high quality oil. Sesame oil is odourless and close in quality to olive oil, straw-like in colour and has an excellent taste (Tunde-Akintunde and Akintunde, 2007). Sesame seed contains antioxidants such as sesamin, sesaminol, sesamol, α and γ-tocopherol (Suja *et al*., 2004; Wan *et al*., 2015), enhancing antioxidant activity in vitamin E in lipid peroxidation which could play vital role in inhibition of development of rancidity (Suja *et al*., 2004), making the oil highly stable especially in hot climates zones. Sesame oil also has additional use in the industrial preparation of perfumery, cosmetics (skin conditioning agents and moisturizers, hair preparations, bath oils, hand products and make-up), insecticides and paints and varnishes. However, all of these uses are comparatively insignificant in terms of the quantities used.

Figure 1: Sesame plant and Seed

Different cultures have different traditional uses for sesame in Nigeria, even though industrial processing and utilization has not been fully developed (Tunde-Akintunde *et al.,* 2012). However, sesame is locally processed and utilized in various forms and the consumption of the oil is increasing worldwide.

The study determined the effect of frying and mode of storage (ambient and refrigeration temperatures) on the quality of sesame oil. The outcome of this research will be beneficial to relevant sesame oil manufacturers as well as consumers, as oils are expected to be of high quality during storage and up to the time of consumption.

MATERIALS AND METHODS

Experimental Materials and Preparations

All chemicals used for this study were of analytical grade. The sesame seeds used for the study were purchased from Garaku market, Kokona LGA, Nasarawa State, Nigeria. The fresh sesame seeds were sorted, dried at room temperature for five (5) days, ground into paste and stored in an air tight container.

Oil Extraction

The oil was extracted manually via cold-press extraction method as described by Kate *et al.* (2014) with slight modifications. The dried sesame seeds were milled in food mill, mixed kneaded repeatedly with intermittent sprinkling of warm water and pressing to remove the oil in without application of any chemical or solvent. The meal was put in a muslin cloth, tied and the oil was pressed out into a container. The oil was settled for two days and then decanted into a container. The oil was centrifuged at 3000 rpm for 3 minutes to separate water and residues from the oil, and then stored in transparent bottles at ambient temperature and refrigerator until analyzed.

Oil Frying

The oil was fried using the method described by Diop *et al.* (2014) with some modifications. Fresh potatoes were washed, peeled, and sliced. The sliced potatoes (830 g) were fried in 1750 ml of freshly extracted sesame oil in an aluminum pot. The pot was heated in a hot plate set at 100 ^{o}C for 60minutes.

Storage studies

The fried and unfried oils were stored in transparent bottles at ambient (30 ^{o}C) and refrigeration (-4 ^{o}C) temperatures for 16 days.

Processing Design

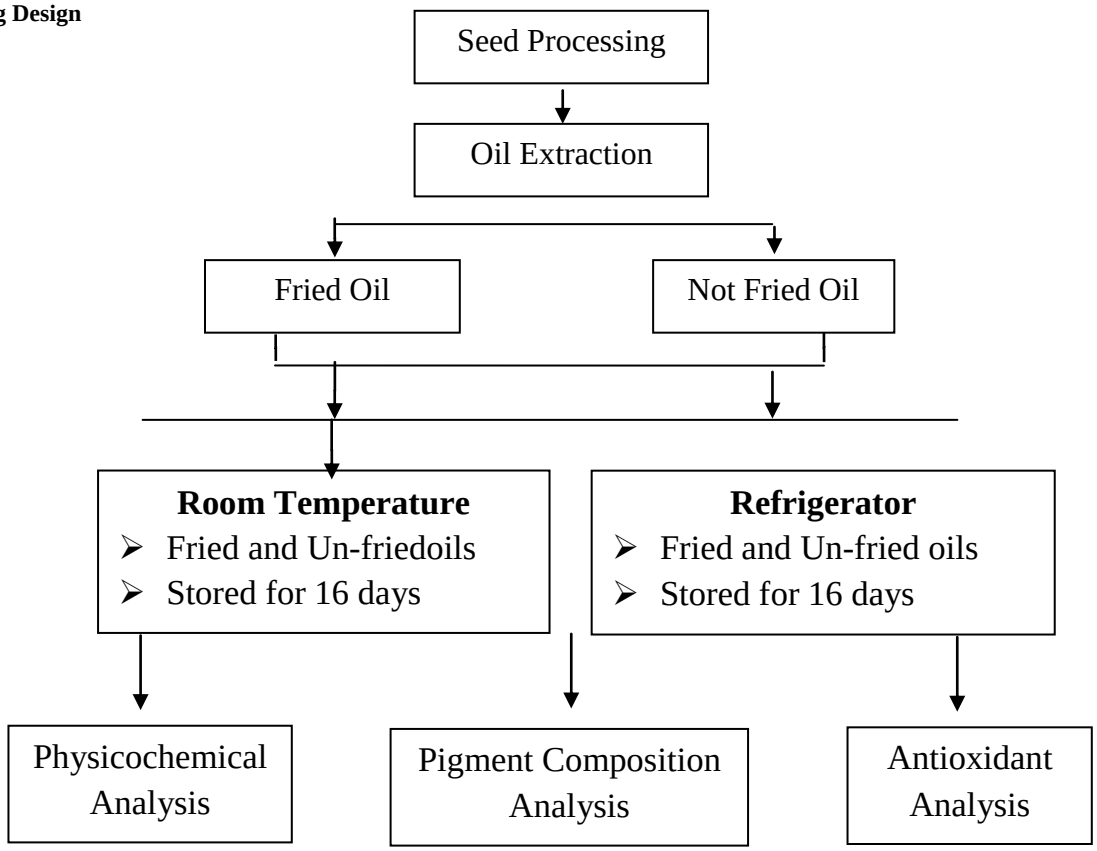

Figure 2: Processing flow chart

Evaluation of Physicochemical Properties

Determination of Density and Specific Gravity

The density was determined by the method described by Garba (2015). An empty beaker was washed dried and weighed using weighing balance. The weight of the beaker was recorded. Exactly 20 cm^3 of each of the oil samples was poured into the beaker and weighed. The weight of the 20 cm^3 of the samples and the beaker was recorded. The procedure was repeated with water and the weight of 50 cm^3 of water was obtained. The density and the specific gravity were calculated as:

Density = $\frac{\text{Weight of Oil Sample}}{\text{Volume of the Oil Sample}}$

Specific Gravity= $\frac{\text{Weight of Oil Sample}}{\text{Weight of Equal Volume of water}}$

Determination of Acid Value (AV)

The acid value was determined by the modified titrimetric method of Pearson (1970) as cited by Anyasor *et al*., 2009). The oil sample (0.5g) was dissolved in the mixed neutral solvent containing equal volumes of diethyl ether and ethanol the indicator (1% phenolphthalein solution) was added and titrated against aqueous 0.1M KOH. The acid value was calculated using the formula:

$$A.V = \frac{\text{Titre Value} + \text{Molar Conc. of KOH} + 56.1}{W}$$

Where

56.1 = Equivalent Weight of KOH

W= Weight of Sample in grams

Determination of Saponification Value (SV)

The Saponification value was determined by the method described by Garba (2015). About 0.5g of the samples were weighed into 250ml conical flasks. 6.2 ml of 0.5N ethanoic potassium hydroxide solutions was added into the conical flasks containing the oil samples with thorough stirring. The reaction mixture was boiled for half an hour until the oil dissolved. The resulting solution was cooled and titrated against 0.5N HCl solution adding 1ml phenolphthalein indicator. The Saponification value (SV) was calculated using the formula:

$$SV = \frac{(B - S) \times N \times 56.1}{W}$$

Where

B= Titre value of blank

S= Titre value of sample

N= Normality of HCl used

W= Weight of Sample in grams

56.1= Equivalent Weight of KOH

Determination of Total Carotenoid Content

The total carotenoid content was determined using the method described by Falade and Oboh (2015). Two (2) mL diethyl ether was added to 0.25mL of the oil sample in a 10mL graduated centrifuge tube. This was saponified with 0.25mL saturated potassium hydroxide (KOH) in distilled water in the dark for 30 minutes. Thereafter, 2.5mL of distilled water was added and centrifuged at 801 ×g for 3minutes. The volume of the ether layer was measured and its absorbance was taken at 450 nm, using diethyl ether as the blank. The absolute value of the total carotenoid was subsequently calculated. The acid value was calculated using the formula :

$$C = \frac{V \times 383 (a_s - a_b)}{100W}$$

Where

C = Carotenoid Content in ppm

V = Volume used for analysis

383= Extinction coefficient

a_s= Absorbance of the sample

a_b = Absorbance of the blank

Determination of Antioxidant Activity Using 2, 2-Diphenyl-1-Picrylhydrazyl (DPPH) Free Radical

The antioxidant activity of the oils extracted from sesame seeds were assayed using DPPH free radical (Brand-Williams *et al*. 1995). To each 0.5 ml test solution, 2.5 ml of 0.1 mM DPPH solution was added. These samples were shaken well and kept in dark for 30 minutes at room temperature. The absorbance was measured at 517 nm against the blank solution consisting 2.5 ml MeOH and 0.5 ml distilled water. The radical scavenging activity was expressed as the radical scavenging percentage as:

$$\% \textit{ Scavenging} = \frac{(As - Ab)}{Ac} \times 100$$

where

As= Absorbance of sample solution,

Ab= Absorbance of blank and

Ac= Absorbance of control

IC50 value was the concentration of sample required to scavenge 50% of DPPH free radical and was calculated from the graph of radical scavenging activity against the concentration of test solution.

Statistical analysis:

Results of four replicate values of each test parameter were pooled and expressed as mean ± standard deviation (STD). One-way ANOVA was performed to evaluate level of significance between the means, Significance was accepted at $p<0.05$

RESULTS

Physicochemical properties

The density and specific gravity of the oil samples after 16 days of storage are presented in Table 1 below.

Table 1: Effect of Frying and Mode of Storage on the Physicochemical Properties of Sesame Oil

	UNREFRIGERATED		REFRIGERATED	
PARAMETERS	**UNFRIED**	**FRIED**	**UNFRIED**	**FRIED**
Density	0.77 ± 0.01	0.76 ± 0.01	0.84 ± 0.02*	0.82 ± 0.01*
Specific Gravity	0.87 ± 0.01	0.85 ± 0.01	0.94 ± 0.02*	0.92 ± 0.01*

Results were expressed in Means ± SD (n = 4).

* = The mean difference is statistically significant across different columns at $p < 0.05$.

The density and specific gravity analysis was carried out on Day 16 and the values are presented in table 1. The result obtained shows that the density and specific gravity of the unrefrigerated oils were significantly higher for the unfried oils (0.77 ± 0.01 and 0.87 ± 0.01) than the fried oils (0.76 ± 0.01 and 0.85 ± 0.01, respectively). The same trend was observed with refrigerated oils. The refrigerated oils had significant ($p<0.05$) higher densities and specific gravities than the unrefrigerated oils. The effect of frying and storage methods on the acid values of the oil samples are presented in Table 2.

The acid values for the unrefrigerated oils were relatively stable from day 4 to day 16 except for Day 12 where there was significant ($p<0.05$) increase in the acid value. This trend was however, different for the refrigerated oils as the values for the unfried oils at Days 12 and 16 (1.15 ± 0.01 and 1.16 ± 0.01mgKOH/g) were significantly ($p<0.05$) high when compared with Day 4 (1.15 ± 0.01mgKOH/g). In contrast, the increase only occurred at Day 16 (1.16 ± 0.01mgKOH/g) for the fried oils at the same mode of storage (refrigeration) condition. The fried oils at both refrigerated and ambient temperature conditions had significantly ($p<0.05$) higher acid values than the unfried oils. The effect of frying and storage methods on the acid values of the oil samples are presented in Table 3. The saponification values increased significantly ($p < 0.05$) with increase in the storage time (day 8 to day 16) although, the oils had high saponification values at Day 4. Generally, there were differences in the saponification values of fried and unfried oils, with the fried oils having higher values. The saponification values of the refrigerated

oils were significantly ($p<0.05$) lower at days 8 and 16 than those of the unrefrigerated oils.

The effect of frying and storage methods on the carotenoid contents of the oil samples are presented in Table 2.The carotenoid contents of the unrefrigerated oils decreased significantly ($p<0.05$) as the storage time increased. However, the carotenoid content of the refrigerated oils was stable throughout the storage period. Generally, the fried oils had significantly ($p<0.05$) lower carotenoid content than the unfried oils. The refrigerated oils had higher although, not significantly different ($p<0.05$), carotenoid content than the unrefrigerated oils. The effect of frying and storage methods on the antioxidant activity of the oil samples are presented in Table 5. The DPPH radical scavenging activities of the fried oils at both refrigeration and ambient temperature storage were higher than the oils that were not fried. The fried oils had low IC50 values when compared to the oils that were not fried. The oils stored at ambient temperature exhibited higher antioxidant activity than the oils that were refrigerated.

Table 2: Effect of Frying and Mode of Storage on Acid Value of Sesame Oil

ACID VALUE (mgKOH/g)				
	UNREFRIGERATED		REFRIGERATED	
DAYS	UNFRIED	FRIED	UNFRIED	FRIED
4	1.14 ± 0.01	1.15 ± 0.01	1.14 ± 0.01	1.15 ± 0.01
8	1.14 ± 0.01	$1.15 \pm 0.01_a$	1.14 ± 0.01	$1.15 \pm 0.01_a$
12	1.15 ± 0.01	$1.15 \pm 0.01^*$	$1.15 \pm 0.01^*$	$1.15 \pm 0.01_a$
16	1.15 ± 0.01	1.15 ± 0.01	$1.16 \pm 0.01^*_a$	$1.16 \pm 0.01^*_a$
REF. VALUE		1.50 ± 0.01		

Results were expressed in Means ± SD (n = 4). * = The mean difference is statistically significant along the same column at the 0.05 level.

$_a$ = The mean difference is statistically significant across different columns at the 0.05 level.

Table 3: Effect of Frying and Mode of Storage on Saponification Value (mgKOH/g) of Sesame Oil

	UNREFRIGERATED		REFRIGERATED	
DAYS	UNFRIED	FRIED	UNFRIED	FRIED
4	111.08 ± 1.97	$181.34 \pm 9.23_a$	$228.47 \pm 2.69_a$	$259.74 \pm 4.37_a$
8	$74.19 \pm 3.31^*$	$95.39 \pm 2.52^*_a$	$88.36 \pm 2.07^*_a$	$123.28 \pm 6.80^*_a$
12	$137.87 \pm 5.64^*$	$152.45 \pm 3.76^*_a$	$125.10 \pm 3.66^*_a$	$135.20 \pm 1.83^*_a$
16	$152.87 \pm 6.19^*$	$159.60 \pm 2.53^*_a$	$143.20 \pm 2.52^*_a$	$149.09 \pm 4.38^*_a$
REF. VALUE		180 ± 3.24		

Results were expressed in Means ± SD (n = 4).

* = The mean difference is statistically significant along the same column at the 0.05 level.

$_a$ = The mean difference is statistically significant across different columns at the 0.05 level.

Table 4: Effect of Frying and Mode of Storage on Carotenoid Contents of Sesame Oil

TOTAL CAROTENOID (ppm)				
	UNREFRIGERATED		REFRIGERATED	
DAYS	UNFRIED	FRIED	UNFRIED	FRIED
0	1.56 ± 0.07	$1.29 \pm 0.06_a$	1.61 ± 0.07	$1.26 \pm 0.09_a$
8	$1.31 \pm 0.03^*$	$1.03 \pm 0.09^*$	1.50 ± 0.03	1.22 ± 0.03
16	$1.33 \pm 0.07^*$	$1.13 \pm 0.03_a$	1.50 ± 0.03	$1.20 \pm 0.07_a$

Results were expressed in Means ± SD (n = 4)

* = The mean difference is statistically significant along the same column at the 0.05 level.

$_a$ = The mean difference is statistically significant across different columns at the 0.05 level.

Table 5: Effect of Frying and Mode of Storage on the Antioxidant properties of Sesame Oil

FREE RADICAL SCAVENGING CAPACITY (%)					
	UNREFRIGERATED		REFRIGERATED		
CONC (mg/ml)	UNFRIED	FRIED	UNFRIED	FRIED	STANDARD
5	35.26	43.08	43.57	46.25	72.48
10	42.67	58.96	49.76	51.30	87.30
15	63.44	68.40	59.61	55.62	88.93
IC50	11.00	7.31	9.40	8.88	- 10.00

Results were expressed in Means ± SD (n = 4)

DISCUSSION

Physicochemical Properties

The physicochemical properties analyzed were density, specific gravity, acid value and saponification value of the sesame oil. According to Yahaya *et al.* (2012), specific gravity is commonly used in conjunction with other parameters such as density to assess the purity of oil (Mestri, 2016). Table 1 shows that the unfried oils had higher density and specific gravity than the fried oils. Free fatty acids may have been released probably due to thermal decomposition of the oil. This probably increased the concentration of the free fatty acids in the oils which eventually lowered the density and specific gravity of the fried oils. The result suggested that the unfried oils had higher purity than the fried oils. The refrigerated oils had higher density and specific gravity than the unrefrigerated oils, indicating that storage at refrigeration maintained the purity of sesame oil. The result was also in agreement with the findings of Fakhri and Qadir (2011), who reported that the density of seed/vegetable oils is dependent on their fatty acid composition, minor components and temperature. Generally, the sesame oil was less dense than water, suggesting its use in cream production as the oil would float and spread easily on the skin (Oyeleke *et al.,* 2012). Acid value is used as an indicator of edibility of oil and its suitability for industrial use (Aremu *et al.,* 2006). The acid value is a measure of the amount of free fatty acid (FFA) in oil, hence it measures the level of hydrolytic rancidity of oils (Gibson, 2018). Table 2 shows that the fried oils had higher acid values than the unfried oils. This increase may be due to thermal hydrolysis of triglycerides which results in the release of free fatty acids in the oil and possible oxidative rancidity. Low acid value of oil indicates that the oil was stable over a long period of time and was protected against rancidity and peroxidation. High acid value, on the other hand, indicates that the oil may not be suitable for use in cooking (edibility). However, such oil will be useful for production of paints, liquid soap and shampoos (Akintayo, 1997; Aremu *et al.,* 2006). Elevated FFA and its oxidation is implicated in aetiology and pathogenesis of some ailments (Jacome-Sosa and Parks, 2014) such as hypertension, Diabetes and others. The Table 2 also shows that the mode of storage had little or no effect on the acid value of sesame oil. The acid values of the sesame oils were low compared with the standard value of 1.50 ± 0.01 (mg KOH/g) reported by Herchi *et al.* (2016), indicating that sesame oil is suitable for consumption.

Saponification value is the amount/mass of potassium hydroxide (KOH) required to neutralize the fatty acids contained in a gram of material (Barret, 2018). Table 3 shows that the fried oils had higher saponification values than the unfried oils. This may be attributed to thermal oxidation that occurred during frying. According to Tunde-Akintunde *et al.* (2012), the high saponification values also suggest the use of the sesame oil for production of liquid soap, shampoos and lather shaving creams. The result obtained also suggested that storage at refrigeration temperature did not adversely affect the saponification value of the sesame oil. The saponification values of the sesame oils were considerably low compared to the standard reference value (180 ± 3.24 mg KOH/g) reported by Herchi *et al.* (2016), indicating that sesame oil may be used more for edibility purpose than industrial purposes.

Carotenoids content

Carotenoids are unsaturated, lipid soluble pigments that contribute to the yellow and orange colour of fruits, oils and vegetables. Table 4 shows that fried oils had significantly ($p<0.05$) lower carotenoid content than the unfried oils. The result implied that carotenoids content of the sesame oil may be affected by oxidation during frying. Rodriguez-Amaya (1999) noted that carotenoids are susceptible to oxidative degradation and isomerization to cis-isomer. He also stated that heat is among the factors that stimulate these processes to occur. The carotenoid values obtained for sesame oil were slightly higher than the value of Coconut oil (0.34 ± 0.1 ppm) and soybean oil (1.39 ± 0.1 ppm) reported by Barnaby *et al*. (2018). The result also shows that oils stored at ambient temperature had lower carotenoid content than the refrigerated oils. This suggests that storage at refrigeration condition would stabilize the total carotenoids in the oil.

Antioxidant Properties

The high radical scavenging capacity and low IC50 values of the fried oils indicate that they have higher antioxidant activity when compared with unfried oils. Deep frying is a complex process involving the oil and the food to be fried. Contact of frying oil with air leads to autoxidation and the formation of large numbers of degradation products which may have antioxidant activity. Thermal stability and increased radical scavenging activity of some oil and essential oils at temperature up to 180 ^{o}C have been documented (Alavi *et al.,* 2010).

CONCLUSION

Based on the results of this study, it is concluded that, Frying decreased the physicochemical properties, the carotenoid content and increased the acid value, saponification value and the radical scavenging activity of sesame oil in both unrefrigerated (ambient) and refrigerated while freezing improved the physicochemical properties, acid value, carotenoid, antioxidant activity but not saponification value (from day 12 to 16) in both unfried and fried sesame oil. It is clear from this work that fried sesame oil stored at ambient temperature may withstand oxidative rancidity better than the refrigerated oil probably due to the presence of favourable breakdown products at ambient temperature as evident in the result of radical scavenging activity of the fried sesame oil stored at ambient temperature. Refrigeration may have conferred rigidity on the molecules of phytochemicals in the sesame oil making it more stable. However, frying may impose detrimental effect on the quality of sesame oil, and can result to gradual loss of bioactive components, including carotenoids, thereby reducing its nutritive value and health benefits. It can also be concluded that sesame oil apart from being used for cooking purposes, soap making, and cream production, could be a potential source for useful antioxidant. Based on the saponification values, sesame oil could be useful in soap making if heated oil is stored for not more than four days.

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