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Impact of Frying Process on Physicochemical Indices of Edible Vegetable Oils in Libya with Varying Omega Fatty Acid Content

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Abstract

The high temperatures employed during the frying process, in the presence of oxygen and water, induce significant chemical transformations in oils, including oxidation, polymerization, cyclization, and hydrolysis. It has been observed that the frying temperature, type of frying oil, presence of antioxidants, and the type of fryer used can impact the extent of hydrolysis, oxidation, and polymerization of the oil. Hence, the objective of this research was to investigate the effects of the frying process on various physicochemical indices of edible vegetable oils with differing omega fatty acid contents. In this study, we focused on assessing key physicochemical characteristics that can serve as quality control parameters for commonly available edible vegetable oils in Libyan markets, namely virgin olive oil, corn oil, sunflower oil, and flaxseed oil. These oils were individually subjected to frying at 180°C for 10 minutes, repeated for three consecutive cycles. The fresh oil, as well as the oils after the first, second, and third frying cycles, were analyzed. The findings indicated that all the examined parameters (free fatty acid percentage, peroxide value, ρ-anisidine value, totox value, and thiobarbituric acid reactive substances) exhibited an increase with prolonged heating time. Among the oils tested, flaxseed oil demonstrated the highest values for the analyzed parameters, followed by sunflower oil, then corn oil, and finally olive oil after three successive frying cycles.

Keywords: olive, corn, sunflower, flaxseed oils, frying process

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Introduction

In the last decades, fast food consumption has become increasingly popular throughout the world (Brown *et al*., 2015 Wang *et al*., 2016). Low cost, desirable taste, convenience, and quick preparation are among reasons that people show tendency towards fast food consumption (Brown *et al*., 2015). Frying, including deep-frying, is one of the most favorable and popular methods of food preparation at home and in industries because of the desirable flavor, texture, and color that it produces in foods (Choe and Min 2007 and Zhang *et al*., 2012). They are extracted from oil containing seed, fruit, or nut tissues of plants by different extraction methods such as solvent extraction, expelling, or combination of these. In some instances, vegetable oils are subjected to physical or chemical refining processes to enhance sensory properties (Mohamed *et al*, 2016). Vegetable oil quality refers to the inherent characteristic of oil including physical, chemical, and nutritional aspects that shall ensure the safety of the oil for consumption. Although there is no official set of standards for assessing edible vegetable oil quality, acid value, peroxide value, and ρ-anisidine value are generally used as quality parameters to determine the oil quality (Oklahoma State University 2016).

Deep-fat frying is a popular cooking technique in the world and vegetable oil is widely used as the frying medium in this process. Deep-fat frying produces both desirable and undesirable compounds through various chemical reactions. Undesirable chemical compounds are formed mainly through hydrolysis, oxidation and polymerization reactions. The frying in oil has become one of the most favored procedures for the culinary preparation as the process is easy, rapid, relatively energy saving, and the resulting meal has an agreeable and intensive flavor. Frying of food is routinely practiced at house-hold level and by the food industry for preparation of fried products. Several vegetable oil are recommended for use in frying. It is a cheap and fast process of simultaneous heat and mass transfer that changes the sensory and nutritional characteristics, as result of complex interactions between food and oil (Ziaiifar *et al*., 2008). The oil which the food is immersed, acts like a heat transferring compound. The process has a preserving action caused by thermal destruction of microorganisms, enzymes and reduction of water activity on the surface of the food. The frying process Simultaneous mass and heat transfer by hot oil modifies the food surface, forming a crust that preserves flavors and retains part of the juiciness of the food while it is cooked, making chewing and digestion easier (Ngadi and Xue, 2009). The rate of heat transfer is influenced by the composition of the food and its properties of heat and mass transfer, including thermal conductivity, thermal diffusivity, specific heat and density. These characteristics change during the frying process, once oil and food are altered. Besides, there are other changes caused by interactions between food compounds (Ngadi and Xue, 2009). One of the most commonly used procedures for preparation and manufacturing of foods throughout the world is deep-fat frying in which the food is subjected to an elevated temperature of 150-190 ^{o}C (Choe and Min 2007). Contact among food, frying oil, and air at high temperatures during deep-fat frying leads to many chemical reactions, thereby affecting the texture, flavor, shelf life, and nutritional quality of both fried food and frying oil in a desirable and undesirable manner (Mohamed *et al*. 2016). Frying is useful in the cooking of all types of foods, such as meat, fish and vegetables. However, potato is probably the food most subjected to frying, since potatoes are used to produce French fries and potato crisps. French fries contain between 8 and 15% fat. In contrast, potato crisps contain rather high amounts of fats, up to 35% (Saguy and Dana, 2003).

An increase in the number of frying cycles decrease triglyceride content and increase polar compounds such as short-chain polymerized compounds and free fatty acids (Tompkins and Perkins 2000). Accumulation of these products lowers the nutritive value and sensory appealing of the vegetable oils. Additionally, free fatty acids in frying oil can accelerate the thermal oxidation of oils (Frega *et al.* 1999). Even though saturated fatty acids provide greater stability in deep-fat frying, they may cause adverse health effects to the consumer (Dauqan *et al*. 2011). However, unsaturated fatty acids are more susceptible to hydrolysis, oxidation, and polymerization reactions and they tend to convert the cis form into transform during the process. Trans fats exhibit proven negative consequences including obesity, cardiovascular diseases, and cancers (Frega *et al*. 1999). Repeated frying causes several oxidative and thermal reactions which results in change in the physicochemical, nutritional and sensory properties of the oil. During frying, due to hydrolysis, oxidation and polymerization processes the composition of oil changes which in turn changes the flavor and stability of its compounds. During deep frying different reactions depend on some factors such as replenishment of fresh oil, frying condition, original quality of frying oil and decrease in their oxidative stability (Choe and Min, 2007). Atmospheric oxygen reacts cause structural degradation in the oil which leads to loss of quality of food and is harmful to human health. Foods fried at the optimum temperature and time have golden brown color, are properly cooked, crispy, and have optimal oil absorption (Blumenthal.,1991). However, under-fried foods at lower temperature of shorter frying time than the optimum have white or slightly brown color at the edge, and have un-gelatinized or partially cooked starch at the center. The under-fried foods do not have desirable deep-fat fried flavor, good color, and crispy texture. Over-fried foods at higher temperature and longer frying time than the optimum have darkened and hardened surfaces and a greasy texture due to the excessive oil absorption (Hassan *et al*., 2014). Oil quality attributes such as FFA, PV, p-AV, TOTOX, and TPC, have been widely used to assess the quality and safety of both fresh and used oils because of the chemical alterations occurred during frying (Karimi and Wawire, 2017). The PV is commonly used as an indicator to assess the oxidative state of oils. This parameter indicates primary oxidation, but it is unstable because peroxides are easily degraded at high temperature (Park and Kim, 2016). Meanwhile, p-AV shows the secondary oxidation products of hydroperoxides, i.e., unsaturated aldehydes, which are more stable than primary oxidation products. The TOTOX value measures the total oxidation products, including both primary and secondary products (Sebastian *et al*., 2014).

Despite the widespread use of the frying process, research on the thermal aspects of frying has been limited. An understanding of the complex processes that occur during frying is necessary to control the quality of the final fried product. The present study, therefore, was conducted to study the effect of frying process of some quality parameters on some edible vegetables oils.

Materials and methods:

Vegetable oils:

Extra virgin olive oil (brand: RS Rafael Salgado, expired date: 8/2024), sunflower oil (brand: El-Amtiaz, expired date: 8/2024), corn oil (brand: El-Baraka, expired date: 12/2024) were purchased from local market in El-Beida city, while flaxseed oil was purchased from herbal shop in El-Beida city. All vegetable oils were of good quality, as indicated by low initial peroxide value, free fatty acid, and low ansidine value.

Potatoes

Potatoes (Solanum Tuberosum) (5 kg) were purchased from a local market in El-Bieda city.

Preparation of potato chips

Potatoes were peeled, cut into 2 mm thick slices using a rotary slicer (Edelstahl, Rostfrel, England), washed and dewatered prior to frying.

Determination of frying optimum temperature and time

The optimum temperature and time of frying of potato chips were determined according to (Barbary *et al.*, 1999). The optimum temperature and frying time obtained for all oils to produce the best quality of potato chips were 180 ºC and 10 min., respectively. The frying was carried out with all samples (Virgin olive oil, corn oil, sunflower oil and flaxseed oil) separately for successive three times. The fresh, first, second and third samples of frying oils were analyzed.

Chemical Analysis of heated oils (Quality Parameters)

The free fatty acid (FFA %) contents were determined by titration with standard potassium hydroxide solution 0.1 N and calculated as oleic acid percentage according to the AOCS official methods (2010) (Method Cd 3a-94).

Peroxide value (PV) was determined according to AOCS official methods (2010) (Method Cd8-53) by titration with standard sodium thiosulphate (0.1 N) and was calculated as milliequivalent oxygen per kilogram oil (meq O_2/kg oil)

Anisidine value was determined colorimetry as described by Egan.*et al.*, (1981) using a spectrophotometer model safas Monaco1900. The anisidine value was calculated using the following equation:

Anisidine value= 25 (1.2 A2-A1) / w

Where

A1= is the absorbance of oil solution.

A2= is the absorbance of reaction product of oil with p-anisidine.

W= is gram of oil present in the 25ml of test solution.

Totox value was calculated according (AOCS.1989) using the PV conjunction with the Anisidine value using the following equation:

Totox value = 2 PV + An V

Where: PV = peroxide value, An V = anisidine value

TBA number was determined according to Allen and Hamilton (1989). The resultant solutions were measured at 538 nm using spectrophotometer. The TBA number was calculated from the following equation:

TBA number = 7.8 D mg malonaldehyde per kg oil

Where: D is the absorbency against blank at 538 nm.

Results and discussion:

Table (1) presents the effect of frying time on the free fatty acids (FFA) content of the four oils during the frying process after three successive cycles. Generally, the FFA percentage increased with longer heating time, but all values remained below 2.0%. This parameter is commonly used to assess the suitability of frying oils for human consumption, with a limit of 2% being defined as the threshold for oil rejection (Matthaus, 2006). The results indicated that flaxseed oil exhibited the highest FFA percentage (1.9%) during the heating process. Free fatty acids (FFA percentage) serve as a measure of the hydrolyzed fatty acids on the triacylglycerol backbone. They are used as chemical markers for monitoring the quality of frying operations (Stier, 2004). Frying oil undergoes degradation during food processing, and FFA percentage is often measured to determine the relative stability of oils towards oxidative and thermal deterioration under deep frying conditions.

Table (1): Effect of frying time on free fatty acids (FFA) % as oleic acid during frying potato chips after three successive time.

Frying period	**Free fatty acids (FFA) % as oleic acid**			
	Olive oil	**Corn oil**	**Sunflower oil**	**Flaxseed oil**
Fresh sample	0.5	0.2	0.42	0.6
1st frying	0.8	0.6	0.9	0.9
2nd frying	1.2	0.9	1.5	1.4
3rd frying	1.4	1.3	1.7	1.9

The maximum allowed value of FFA varies depending on the type of food being fried and number of batches during frying operation (Shaker 2014). Gunstone (2008) suggested that the FFA content of the refined oil should be less than 0.1% because most of the FFAs from crude oil are removed during the refining process.. Sebastian *et al.* (2014) have found that the FFA levels in fresh oil samples varied from 0.05 to 0.08% and, for in-use samples from the fryer, ranged widely from 0.25 to 3.99%. The increase in FFA value is not an unswerving parameter for degradation of frying oil because it is difficult to differentiate between FFA formed by oxidation or by hydrolysis (Frega *et al.* 1999; Mohamed *et al.* 2006).

Table (2) presents the effect of frying time on the peroxide value (PV) of the four oils during the frying process after three successive cycles. Generally, the PV increased with longer heating time, but all PV values remained below 10 meqO_2/kg oil. The results also revealed that flaxseed oil exhibited the highest PV value during the heating process, which can be attributed to its initially higher

Table (2): Effect of frying time on Peroxide value (PV) (meqO_2/kg oil) during frying potato chips after three successive time

Frying period	Peroxide value (PV) (meq O_2/kg oil)			
	Olive oil	Corn oil	Sunflower oil	Flaxseed oil
Fresh sample	0.571	0.627	0.687	1.35
1st frying	4.98	4.85	4.28	9.85
2nd frying	5.63	6.14	6.34	7.98
3rd frying	7.52	8.12	9.51	9.96

PV value (1.35 $meqO_2$/kg oil). The PV for flaxseed oil reached 9.96 $meqO_2$/kg oil after three successive cycles. Additionally, the data demonstrated that PV increased progressively with longer heating time.

The higher ramp rate observed for flaxseed oil can be attributed to its high content of C18:3. These acids provide more active methylene groups, which are more prone to oxidative deterioration (Tyagi and Vasishtha, 1996). These results indicate that the oxidation stages of oils during frying result in the formation of free radicals, which undergo initiation, propagation, and termination stages. The primary products of lipid oxidation are hydroperoxides. Hydroperoxides (ROOH) are the major initial reaction products of fatty acids with oxygen (Burcham, 1998).

Peroxide value (PV) is a useful indicator of oxidation at the initial stage, but it is not related to the frying duration (Atinafu and Bodemo 2011). Cheman and Wanhussin (1998) mentioned that a good-quality vegetable oil should have a PV of lower than 2 $meqO_2$/kg. The primary product of lipid oxidation is hydroperoxide, which is generally referred to as peroxides (Allendorf 2010). PV is influenced by constituent fatty acids and the length or type of storage (Lawson 1995). PV may increase after the sample is taken from the fryer. Therefore, PV is generally not a very reliable parameter to determine deterioration of cooking oil (Man and Hussain 1998). Naz *et al.* (2005) have suggested that the increase in PV of oil follows the order deep frying > air–light exposure > air exposure. Yılmaz *et al.*, 2023 studied the Deep frying processes (180 ± 5°C) carried out with a total of one liter of each type of oil and 100-g potatoes at every turn (sliced into 1 cm * 1 cm * 6 cm pieces). The process was carried out keeping the conditions constant for all oils and was repeated 10 times consecutively for all oils. They found the peroxide value for some kinds of oils of base line, 5th frying and 10th frying for refined olive oil were: 7.3, 7.6 and 12.0; for extra virgin olive oil were: 3.8, 8.2 and 9.4; for fortified sunflower oil were: 3.6, 5.2 and 9.4; for sunflower oil were: 6.4, 21.0 and 7.4; for corn oil were: 3.0, 7.4 and 16.0 $meqO_2$/kg respectively.

Table (3) presents the effect of frying time on the Anisidine value (p-AV) content of the four oils during the frying process after three successive cycles. Generally, the Anisidine value (p-AV) content increased with longer heating time, P-AV is also defined as the relative amount of nonvolatile unsaturated aldehyde compounds formed by the secondary degradation products from fatty acid hydroperoxide. List *et al.* (1974) were the first researchers to use the p-AV test to judge oil quality and had demonstrated a highly significant linear correlation (r = −0.82) between p-AV and flavor scores in soybean salad oil. Al-Kahtani (1991) reported that the p-AV was the most reliable measure

Table (3): Effect of frying time on Anisidine value (p-AV) during frying potato chips for three successive time.

F Frying period	Anisidine Value (p-AV)			
	Oli olive oil	Corn oil	Sunflower oil	Flaxseed oil
Fresh sample	0.15	0.22	0.4	0.8
1st frying	2	5	8	11
2nd frying	6	9	13	16
3rd frying	12	17	18	19

of oil oxidation and the discarding oil has PV levels in the ranges 14.2 to 55.8. According to Gupta (2005) adesirable p-AV for fresh frying oil should be less than 4.0, with an upper limit of 6.0. He also mentioned that if the fresh oil had a p-AV above 6, the oil would be highly oxidized. The secondary products of oxidation are more stable than the unstable peroxides formed in the primary stages of the oxidation. Results again revealed that flaxseed oil showed the highest p-AV value during the heating process. The (p-AV) reached 19 for flaxseed oil for three successive times as compared to other oils. Results also revealed that the longer the heating time, the higher the (p-AV) observed.

The totox value is often considered to have the advantage of combining evidence about the past history of oil (as reflected in the p-AV) with its present stage (as evidenced in the PV). Therefore, determination of totox value has been carried out extensively to estimate oxidative deterioration of food lipids (Rossell 1983). This value represents the oil or fat quality, oxidation status and presence of degradation products formed from previous oxidation of oil, totox value during the frying of potato chips after three successive cycles. The results revealed a similar trend as observed in the determination of FFA% and PV, where flaxseed oil exhibited a high totox value (39.92) after three successive cycles (Table 4). Meanwhile, olive oil recorded the lowest totox value compared to the other oils. Kajimato et al., 1996 confirmed a significant correlation between PV and An-V, as well as the stability of the oil, to assess the deterioration that occurred in the frying oils. These findings are consistent with the observations made by Hansen et al. (1994), who reported an increase in both peroxide and Anisidine values during the frying process.

Table (4): Effect of frying time on totox value during frying potato chips after three successive time.

Frying period	totox value			
	Olive oil	**Corn oil**	**Sunflower oil**	**Flaxseed oil**
Fresh sample	1.292	1.474	1.774	3.5
1st frying	11.96	14.7	16.56	30.70
2nd frying	17.26	21.28	25.68	31.97
3rd frying	27.04	33.24	38.02	39.92

Table (5) presents the effect of frying time on the TBA number of the four oils during the frying process after three successive cycles. Generally, the TBA (mg malonaldehyde/kg oil) increased with increasing heating time for all oils after three successive cycles.

Flaxseed oil exhibited the highest TBA value, followed by sunflower oil, corn oil, and finally olive oil. As the TBA value is a measure of oxidation, these results were

Table (5) Effect of Frying time on TBA value (mg malonaldehyde/kg oil) during frying potato chips for three successive time

Frying period	TBA value (mg malonaldehyde/kg oil)			
	Olive oil	**Corn oil**	**Sunflower oil**	**Flaxseed oil**
Fresh sample	2.2	2.88	3.00	3.3
1st frying	2.9	3.12	3.5	4.00
2nd frying	3.4	4.0	4.1	4.4
3rd frying	4.2	4.8	4.9	5.1

expected. Flaxseed oil, which had the highest oxidation rate among the three studied oils, also had the highest TBA values.

Since oil undergoes a variety of chemical changes during cooking and frying, the chemical changes brought about by heating are extremely important from both nutritional and toxicological perspectives for both consumers and the processing industry (Chung and Shu-yueh, 1993). Malonaldehyde is a relatively minor product of the oxidation of polyunsaturated fatty acids, which reacts with the TBA reagent to produce a pink complex with an absorption maximum at 530–532 nm (Tarladgis *et al.*, 1964). According to Hassanien and Sharoba (2014), the TBA value is typically low for oil samples derived from continuous frying due to the low concentrations of frying oil in fatty acids with more than two double bonds, which are the main precursors of MDA. Similar results were reported by Sebedio *et al*. (1991) for palm oil.

Conclusions

The results obtained from the study demonstrate that the FFA% (free fatty acids), PV (peroxide value), P-An (ρ-Anisidine value), TOTOX value, and TBA (thiobarbituric acid reactive substances) increased with the prolongation of the frying process time. However, it is important to note that all the analyzed parameters remained within the permitted values. These findings indicate that the studied parameters can serve as reliable indicators for assessing the extent of lipid deterioration during the frying process.

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المجلــة الليبيــة للـغـذاء والتـغذيــة
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تأثير عملية القلي على المؤشرات الفيزيائية والكيميائية للزيوت النباتية الصالحة للأكل في ليبيا مع تباين محتوى الأحماض الدهنية أوميجا

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قسم الكيمياء، كلية العلوم، جامعة عمر المختار، البيضاء

المستخلص

تؤدي درجات الحرارة المرتفعة المستخدمة أثناء القلي في وجود الأكسجين والماء إلى تغييرات كيميائية مهمة في الزيوت عن طريق الأكسدة والبلمرة وتكون مركبات حلقية والتحلل المائي، وقد وجد ان هناك تأثير لدرجة حرارة القلي ومضادات الأكسدة في زيت القلي ونوع المقلاة على التحلل المائي والأكسدة وبلمرة الزيت أثناء القلي. لذلك كان الهدف من هذا العمل دراسة السلوك الحراري للزيوت النباتية المختلفة التي تحتوي على أحماض دهنية مخنلفة من الأوميغا. خلال هذا البحث تمت دراسة العديد من الخصائص الفيزيائية والكيميائية والتى يمكن استخدامها لمراقبة جودة الزيوت النباتية الرئيسية الصالحة للأكل المباعة في الأسواق الليبية وهي زيت الزيتون البكر وزيت الذرة وزيت عباد الشمس وزيت بذور الكتان وتم قليها عند 180 درجة مئوية لمدة 10 دقائق بشكل منفصل ثلاث مرات متتالية. تم تحليل العينات الطازجة، الأولى، الثانية والثالثة من زيوت القلي. أظهرت النتائج أن كل من، قيم الاحماض الدهنية الحرة والبيروكسيد والبارانيسيدين والتوتكس وحمض الثيوباربيوتريك قد زادت مع زيادة زمن التسخين، وأظهر زيت بذور الكتان قيم عالية كذلك لنفس الخصائص يليها زيت عباد الشمس ثم زيت الذرة وأخيراً زيت الزيتون بعد ثلاثة مرات متتالية من القلي.

الكلمات المفتاحية: زيت الزيتون، الذرة، عباد الشمس، زيوت بذور الكتان، عملية القلي

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