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Changes in physicochemical properties and kinetics oxidation of refined sunflower oil stored in packaging materials commonly used in Egypt

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Abstract

The packaging materials polyethylene (PE), Glass (G) and polyethylene terephthalate (PET) are considered the most commonly used in packing edible oil in Egypt. So, the current investigation aimed to study the Changes that occurred in physicochemical properties, fatty acid composition, Atherogenicity index (AI) factor, C18:2/C18:3, total saturated fatty acids/total unsaturated fatty acids (TSFA/TUFA) ratios and oxidative stability (OS) of refined sunflower oil (RSO) as a result of its storage at room temperature for 6 months in three different types of packaging materials (PE, G and PET). The OS (induction period, IP) of stored RSO were estimated using Rancimat at three different temperatures being 90,100 and 110° C. The obtained data of these tests were used in estimating the thermo dynamic properties such as Activation energy (Ea), Arrhenius factor (Arrh.Factor), activation enthalpies (Δ H) and entropies (Δ S). As well as the Shelf-lives (predication index IP₂₅) of investigated oil samples have been also predicted. The results showed that OS of RSO stored in different packaging materials was in the order; control >PET > G > PE and vice versa for values of kinetic parameters (Ea, Arrh.Factor, Δ H & Δ S), whereas the values of oxidation kinetic parameters were in the order PE >G>PET> RSO control. Also, the results observed that IP₂₅, for the investigated oil samples were decreased in order to; control > G > PET > PE.

Keywords: Packaging materials, Storage, Refined sunflower oil, Fattyacids composition, Rancimat, Kinetic parameters and Oxidative stability.

1. Introduction

Egypt uses over 2.5 million tons of edible oils each year. Of this total, only 48.5 thousand tons are produced locally at extraction factories by the extraction of oilseeds. Cottonseed, a byproduct of the cotton ginning business, accounts for almost half of this quantity produced. In addition to the remaining half is extracted from both imported soybean and localsunflower oilseed. This means that only 2% of the country's total edible oil consumption is met by the oil generated in Egypt through the extraction of both domestic and foreign oilseeds. Imported oils have been used to cover the remaining 98% [1].

Sunflower, soybean, and cottonseed oils are the three main sources of vegetable oils in Egypt. As a result, they are employed in cooking, frying, and salad dressings. One of the significant oilseeds that are grownextensively worldwide is sunflower.

It is among Egypt's top two or three significant oily crops. Sunflower is regarded as the world's fourth-largest source of vegetable oil[2]. Sunflower oil has the greatest consumption position among edible oils. For a number of factors, including its lower cost compared to other edible oils and its flavor and smell that most consumers find agreeable [3].

*Corresponding author e-mail: dr_hisham_nrc@yahoo.com.; (Hisham A. Abd El-lateaf). Received date 23 June 2024; revised date 01 August 2024; accepted date 03 August 2024 DOI: 10.21608/EJCHEM.2024.298867.9889 ©2025 National Information and Documentation Center (NIDOC) Alpha-tocopherol is present in sunflower oil and is a significant source of vitamin E. Its content of linoleic acid (C18:2, ω 6) is high. This unsaturated fatty acid's high reactivity is linked to oxidation. The modest extent to which oxidation occurs in sunflower oil makes the loss of nutritional value and quality particularly interesting [4].

The degree of saturation, naturally occurring or added antioxidants, pro-oxidants, or previous misuse all affect an oil or fat's ability to oxidation resistance. Once this resistance is overcome, oxidation begins to accelerate and become extremely rapid. Until then, oxidation remains gradual. The resistance to oxidation is determined by the duration of time preceding this abrupt increase of oxidation, which is known as the induction period [5]. Storage containers not only should shield the oil from air, light, and humidity, but they should also prevent any chemical interactions with the oil phase that could change the oil's safety and quality [6]. The main factor causing quality losses in crude and refined oil during storage is autoxidation, also known as oxidative rancidity. The degree of process, initial composition, concentration of minor compounds with antioxidant or pro-oxidant properties, and storage conditions all affect how oils oxidatively stabilize and deteriorate [7]. Sunflower oil packaging is crucial. The Physical characteristics of packaging material may directly impact on the oil's quality. Oil is packaged using glass, metals, and various polyethylene types (bags). Glass has many benefits, including stiffness and inertness, but it is also expensive and brittle, which is why molded thermoplastic containers have been developed [8]. Packaging materials are including PE and PET. PET is rapidly gaining popularity due to its superior mechanical qualities, low weight, low price, and excellent barrier. Conversely, a significant drawback of plastic packaging is that polymers enable mass transportation [4].Rancimat can be used to lower temperatures (e.g., 25 °C) as IP25 to predict the shelf-life of oils in a few of hours [9].

The Arrhenius equation and activated complex theory were used to estimate Ea, Δ H and Δ S which varied from 92.05 to 99.17 kj /mol, 88.83 to 95.94 kj/mol and – 35to – 4.81 kj /mol respectively [10]. Oil with a lower Ea value would have a slower rate of lipid oxidation and a later start to rancidity. According to [11], oil with lower Δ H and Δ S levels had slower rates of lipid oxidation. These values showed a strong correlation with those of similar unsaturated plant oils [12]. The negative values of Δ S of oil indicated the production of an ordered activated complex, which is preferred for increased resistance to oxidation [9]. The oxidation kinetics (K) showed an increasing trend as a function of temperature (100–130°C). According to higher K values, higher values suggested faster oxidation [10]. The increased use of plastic containers for packaging edible oils in Egypt motivated the present study to compare the effect of packaging containers to keep the quality of sunflower oil. In this study sunflower oil was packaged in both PE, G and PET containers and storage at room temperature for 6 months. Then the changes in physicochemical properties, fatty acid composition, OS that occurred in RSO as well as kinetic oxidation parameters were estimated. Based on these results can be identify the best type among the three former containers that can be used for filling the RSO in the Egyptian market.

2. Materials and Methods

2.1 Materials

Refined sunflower oil (RSO) without any addition of antioxidants, was provided from local factory located in, El Sadat city, Menoufia, Egypt.

Packaging materials; polyethylene (PE), glass (G) and polyethylene terephthalate (PET) containers obtained from local market in Cairo, Egypt.

Sampling:

RSO was filled into 0.5-liter containers after being sampled in accordance with method of [13].

Storage conditions:

The RSO was filled to the brim in the G, PE, and PET containers, which were hermetically sealed with very little space between the cap and the product.

Following bottling and sealing, the samples were maintained under carefully monitored conditions in a dry, room-temperature environment with artificial lighting.

2.2 Methods

2.2.1 Physico-Chemical characteristics of oil samples

Egypt. J. Chem. 68, No.3 (2025)

Refractive index RI, color, acidity (FFA% as oleic acid) and peroxide value (PV) were determined according to the method of [14].Iodine (IV) and saponification(SV) values of RSO were calculated from fatty acidspercentage by equation according to [15].

2.2.2. Determination of fatty acid composition of oil samples 2.2.2.1. Preparing of fatty acids methyl esters

The fatty acids methyl ester of oil samples was prepared using trans-esterification with cold methanolic solution of potassium hydroxide according to the method of [16].

2.2.3. Gas liquid chromatographic analysis of fatty acid methyl esters

The identification of the components of fatty acids methyl esters were done using an Perkin Elmer Auto System XL gas chromatograph equipped with flame ionization detector (FID),Fused silica capillary column ZB-Wax (60 m x 0.32 mm i.d). One microliter of FAME mixture was injected into the GC system. The inlet temperature was 240°C and the split ratio was 10:1. The carrier gas was helium, flow rate at 1 ml/min constant flow. The oven temperature was programmed at initial 50°C, held for 1 min, then programmed from 50 to 220°Cat rate 3°C /min, then held at 220°C for 20 min. The detector was set at 250°C with 450 ml/min air flow, 45 ml/min hydrogen flow.Fatty acid methyl esters were identified by comparing their relative and absolute retention times to those of the authentic pure standards of fatty acid methyl esters.

2.2.4 Determination of AI factor

Atherogenicity index (AI) is an indicator for oil stability. The increasing in AI factor is considered harmful to health, and calculated from fatty acids composition as predictors according to [17].

AI = [C12 + 4 (C14) + C16] / TUFA(total unsaturated fatty acids).

2.2.5. Determination of the oxidative stability index (OSI) by Rancimat

The induction periods, as the oxidative stability index (OSI), of the investigated oil samples was measured by (professional automated RancimatMetrohm Co., Switzerland, model 892) at 90. 100 and 110 ± 0.2 °C with an air flow rate of (20 L/h), using 3g of the tested oils were accurately weighed into each of the 8 reaction vessels. The OSI method is based on determining the time (usually termed "induction time") before the maximum rate change of oxidation by measuring the increase in conductivity of de-ionized water caused by dry air bubbled through a heated sample that carries the resulting volatile acids into a separate container with thede-ionized water according to the method described by [18].

2.2.6. Oxidation kinetics data analysis:

The kinetic parameters were estimated for investigated oils according to the method described by [19]. A kinetic rate constant (k) for lipid oxidation of investigated oils was taken as an inverse of IP(k=1/IP, h-1). Using the Arrhenius equation (Eq. (1)), the activation energies (Ea, kJ/mol) and frequency factors (A, h-1) for lipid oxidation in investigated oils were calculated using:

 $\ln(k) = \ln(A) - (Ea/RT) \dots (1)$

Where k is the kinetic rate constant (h-1), and R is the molar gas constant

(8.314 J/mol K).

Using the activated complex theory (Eq. (2)), the activation enthalpies (Δ H) and entropies (Δ S) of lipid oxidation in investigated oils were calculated using:

 $\ln(k/T) = \ln(kB/h) + (\Delta S/R) - (\Delta H/RT) \dots (2)$

Egypt. J. Chem. 68, No. 3 (2025)

where kB is Boltzmann constant (1.380×10–23 J/K) and h is Planck's constant (6.63×10–34 J s). The Δ H and Δ S were calculated from the slope and intercept of Eq. (2).

Shelf-life prediction:

The shelf-life of investigated oils was predicted by plotting the natural logarithm of IP vs. absolute T (K) using Eq. (3).

 $\ln(IP) = a(T) + b....(3)$

Where a and b denoted slope and intercept of Eq. (3), respectively.

2. Results and discussion

3.1. Physicochemical characteristics of oil samples

The influence of different packaging containers (PE, G and PET) and storage period for six months at room temperature on the physicochemical properties (RI, color, acidity (FFA), PV, IV and SV of RSO were summarized in Table (1). The values of the previous parameters of stored RSO in the mentioned packaging materials indicated that, there were differences in these values as a result storage the RSO in different types of packaging materials.

Table 1

Physical and Chemical properties of RSO stored in different packaging materials

Property	Control	Packaging materials		
		PE	G	PET
Refractive index at 25°C	14651	14600	14620	14630
Color R/Y	0.3/3	0.7/8	0.6/7	0.6/7
Peroxide value	0.00	8.01	7.91	4.43
(m eq O ₂ /kg oil)				
Acidity	0.03	0.73	0.33	0.26
(% as oleic acid)				
Iodine value	138.94	133.80	133.84	134.21
(g of I ₂ /100g oil)				
Saponification value	194.65	199.86	198.79	199.48
(mg KOH/g of oil)				

The results indicated that value of RI decreased, but value of color (R/Y) increased with storage period in all packaging containers compared to control sample. The decrease in RI value may be attributed to the lowering in TUSFA as a cleared in Table (2 and 3). Whereas, RI can be used to identify and estimate the degree of unsaturation, the high concentration of unsaturated fatty acids in an oil causes an increase in RI and vice versa. Additionally, the PV and FFA values for the stored RSO packaged in PE material were found to be 8.01 meq O_2 / kg oil and 0.89 %, respectively. This may be related to the combined effects of the relatively higher PE to oxygen of PE with oil that caused the oil to oxidize quickly. On the other hand, the lowest values for acidity and PV were obtained for the RSO packaged in PET and G containers, which were 0.26 % and 4.43 meq O_2 / kg oil and 0.33 % and 7.9 meq O_2 / kg oil, respectively. The oil packaged in PET containers may have observed a lower increase in PV and FFA levels due to the chemical activity of the container. The decreased rise in PV and FFA values of oil packaged in G containers may also be related to glass's strong O_2 barrier and chemical inactivity [20].

3.2. Fatty acids composition of oil samples

The changes in fatty acids composition of RSO stored in various containers constructed from (PE, G and PET) are shown in Table (2). From the tabulated data there was an increase in value of C16:0, C18:0 and C18:1acids, while the values of C18:2 and C18:3 acids recorded decreases as a result storage in all types of packaging materials compared to control sample.

155

According to the data in the same table, stored samples taken from all containers showed the presence of C14:0, C16:1, C17:0, C17:1, and C20:1 acids in comparison to the control sample. Also, the results showed that RSO stored in PET container had the higher values from C16:0, C18:0 and C18:1 acids as well as total saturated fatty acids (TSFA) compared with their values in RSO stored in the other packaging containers. These results parallel with OS of oil stored in PET container, whereas oil taken

from the same container gave the highest value of OS by storage compared to other container as a shown in Table (3). Also, data revealed that there were changes in (AI) factor, TSFA/TUSFA and C18:2/C18:3 ratios for stored RSO in the same mentioned

previous containers. It was evident from the tabulated data that values of AI factor, C18:2/ C18:3 and TSFA/ TUSFA ratios for stored oils taken from all container recorded increment compared to control sample. This increase in AI factor due to increase amount of C14:0 and C16:0 acids and reduce total unsaturated fatty acids in stored RSO taken from all containers compared to control sample. Diets rich in the C18:2/C18:3 (n-6/n-3) ratio and AI factor are typically thought to be harmful to health [17]. As well as oils with high TSFA/TUSFA ratio is deleterious to health.

Table 2

Fatty acids composition of RSO stored in different packaging materials

Fatty acid (%)		Packaging materials			
	Control	PE	G	PET	
C14:0		0.07	0.07	0.08	
C16:0	6.55	6.93	7.20	7.23	
C16:1		0.13	0.13	0.15	
C17:0		0.24	0.04	0.03	
C17:1		0.03	0.03	0.03	
C18:0	2.89	3.84	3.79	3.87	
C18:1	27.74	28.28	28.73	28.89	
C18:2	61.82	58.91	58.96	58.78	
C18:3	1.00	0.89	0.87	0.89	
C20:0		0.37			
C20:1		0.21	0.18	0.16	
TSFA	9.44	11.45	11.10	11.21	
TUSFA	90.56	88.55	88.90	88.89	
TSFA/TUSFA	0.104	0.129	0.125	0.126	
C18:2/C18:3	61.82	66.27	67.77	66.04	
Atherogenic index	0.072	0.081	0.084	0.084	

3.3. Oxidative stability of oil samples

The OS of RSO at 90, 100 and 110°C stored in different packaging material; (PE, G and PET) containers for six months at room temperature shown in Table (3). It could be demonstrated based on the results in this table that, a reduce in values of OS of RSO at 90, 100 and 110 °C as a result storage period in all types of packaging materials compared to control sample. With respect to RSO sample stored in the PET container showed the lower decrease in OS at 90, 100 and 110 °C (12.59, 6.2 and 3.10 hrs. respectively), while the higher decrease was exhibited for sample in PE materials, were 7.64, 3.68 and 1.72 hr. at 90, 100 and 110°C respectively compared with control sample. The lower drop in OS of RSO oil filled in PET containers may be due to the fact that sunflower oil's quality is greatly influenced by the container's ability to keep out light and oxygen, which further delays oxidative changes. PET showed to be more than sufficient to maintain the sunflower oil's purity and prevent oxidation

for up to a year when kept in the dark [8]. On the other hand, PE packaged materials may be more prone to quality degradation because of their quick deterioration from corrosion and ensuing pitting corrosion, which may be the cause of the oil's quick oxidation in the packaging. The G material had the intermediate decrease in values of OS at the same previous temperature, were 9.66, 4.42 and 2.19 hr., respectively. Either light absorption into the packing materials, oil compound

breakdown as a result of initial oxygen concentration and oxygen permeability through the package, or both aspects could account for the variation in OS values of RSO packaged in previously examined materials [8].

Oxidative stability (Induction period) of RSO stored in different packaging materials				
Oil sample	Induction period (hrs) at			
	90° C	100° C	110° C	
Control	18.32	10.22	5.13	
PE	7.64	3.68	1.72	
G	9.66	4.42	2.19	
PET	12.59	6.2	3.10	

3.4. Shelf-life prediction index (IP25) of oil samples

Data in Table (4) and Fig (1), revealed that the changes in shelf-life by hours, days and months (prediction index IP₂₅) of RSO as a result stored in PE, G and PET containers for six months. From the results the G container had a higher value of IP₂₅ followed by PET then PE containers compared to control. The current findings are consistent with those published by [8], which demonstrated that sunflower oil kept in dark, glass, and PET containers exhibited very little oxidation and kept its original profile for an extended length of time.Finally results observed that IP₂₅, suggesting better thermal stability for investigated samples, which decreased in the order: Control > G> PET > PE.

Table 4

Shelf-life prediction index (IP25) of RSO stored in different packaging materials

Oil sample	Shelf-life predict	Shelf-life prediction index (IP ₂₅)		
	Hours	Days	Months	
Control	1402.48	58.44	1.95	
PE	1150.56	47.94	1.60	
G	1258.91	52.45	1.75	
PET	1211.97	50.50	1.68	



Fig. 1. Semi-logarithmic relationship between ln(IP) and temperature values (T) for Shelf-life (IP₂₅) prediction of investigated oil samples.

*Egypt. J. Chem.***68**, No.3 (2025)

Table 3

3.5. Kinetics oxidation parameters of oil samples

Numerous investigations have demonstrated that oil's kinetic parameters can be determined and assessed using the Rancimat test. Determination these kinetic characteristics are useful for identifying the provenance of different oils, describing the variations or similarities in the oils, and for forecasting the oil's oxidative stability in different storage conditions [19, 21]. As shown in (Table 5) and Figs (2 and 3), the kinetic parameters of lipid oxidation (Ea, Δ H, Δ S) were determined for RSO stored at room temperature for 6 months in different packaging materials containers(PE, G and PET) compared to control. Ea ranged between 73.49 kJ/mol for control and 86.14 kJ/mol for PE containers, which correlated well with related oils [19, 9]. The reduction in Ea values indicate a slower rate of lipid oxidation and, hence, delayed of the initial oxidation reaction [12]. Accordingly, the relative stability of RSO stored in containers made from three types of packaging materials (PE, G and PET) compared to control sample was in the order: PE < G < PET < control. Arrh.FactorA,h-1 values are presented in Table 5 were 3.19x1011, 2.30x1011 and 3.54x1010 for RSO stored with the three different types of packaging materials PE, G and PET respectively, compared to control 2.00x109, which indicated that even modest changes in Ea led to significant changes in Arrh.FactorA,h-1. As shown in Table 5 the values of thermodynamic parameters for lipid oxidation (Δ H and Δ S) for investigated samples were estimated. ΔH ranged from (70.39 to83.04 KJ/mol) for control sample and RSO stored in containers made from packaging material (PE) respectively. As well as, ΔS ranged from (-76.99 to -34.84 KJ/mol) for control and (PE) samples respectively. The changes in ΔH and ΔS were similar to Ea. An inverse relationship was noted between these criteria and IP of investigated samples. As a result, lower kinetic parameter values were found in oil samples with greater IP, characteristics that are preferred for improved thermal stability. The negative values of ΔS showed the formation of an ordered activated complex, which is beneficial for increased stability against oxidation [9].



Fig. 2. Semi-logarithmic relationship between ln(k) and temperature values (1/T) for lipid oxidation of investigated oil samples.

Table 5

Oxidation Kinetic parameters of RSO Stored in different packaging material

	i01 <i>)</i>
Control 73.49 2.00x10 ⁹ 70.39 -76.99	
PE 86.14 3.19x10 ¹¹ 83.04 -34.84	
G 85.79 2.30x10 ¹¹ 82.69 -37.58	
PET 80.99 3.54x10 ¹⁰ 77.89 -53.13	



Fig.3.Semi-logarithmic relationship between ln(k/T) and temperature values (1/T) for lipid oxidation of investigated oil samples.

4. Conclusion

This study can contribute to determining the best type of packaging material among the three types (PE, G and PET) under study that are commonly used in the Egyptian market for packaging edible oil. The study showed the changes in physicochemical properties, fatty acid composition, OS, kinetic oxidation parameters that occurred in RSO as a result of its storage at room temperature for 6 months in these three different types of packaging materials .Also, the study showed that RSO stored in PET container had the highest values of oxidative stability, quality characteristics and kinetic parameters followed by those in the G and PE containers .while, RSO stored in G container had the highest value for IP₂₅ compared to those in the other containers. Furthermore, study indicated that oil samples with higher IP had lower values of the kinetic parameters (Ea, Δ H, and Δ S), which are desirable for better thermal stability. An ordered activated complex formed, as indicated by the negative values of Δ S, which is advantageous for greater stability against oxidation.

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6. Conflicts of interest

No competing interests are disclosed by the authors.

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Egypt. J. Chem. 68, No. 3 (2025)