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Comparative Study of Edible Oils Commonly Used in Egypt in View of Their Physico-Chemical Characteristics and Oxidation Kinetics

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ABSTRACT

The oxidative stability (OS) of the edible oils commonly used in Egypt were compared using rancimat. These include refined sunflower (SF), soybean (SB), corn (CO) and palm olein (PO) oils. The Physico-chemical characteristics of these oils have been identified as well as their fatty acid composition. Their oxidation rates were measured at three different temperatures being 100,110 and 120° C. In view of the results of those tests, the oxidation rate constant; k as well as other thermo dynamic properties such as the Activation energy (Ea), Arrhenius factor (Arrh.Factor), activation enthalpies (ΔH) and entropies (ΔS) were estimated. The Shelf-lives hours, days and months (predication index IP_{25}) of all the tested oil samples have been also predicted.

Keywords: oxidative stability, edible oils, rancimat, Physico-chemical, oxidation rates

1. Introduction

Both the food and the energy crises are pressing issues facing the globe today. These are caused by the world population growing at an exponential pace as well as an increase in the rate of per capita consumption of both food and energy. The number of people on the planet is growing by roughly 75 million each year, or 1.1% annually. From 1800 to 2012, it climbed from one billion to seven billion. It is anticipated that population growth will continue, with estimates of 8.4 billion by mid-2030 and 9.6 billion by mid-2050 (World Population Data Sheet, 2014). On the other hand, Egypt's population is predicted to increase by 1.5 million people year and reach 95.6 million by 2026. (Khalifa *et al.*, 2000). As a result, it appears that the food and energy problems will undoubtedly get worse very soon. There are several well-known oilseeds in the globe that fall into two primary categories. Oilseeds are included in the first category because they may provide a safe supply of oils for human consumption. The other group, on the other hand, has the potential to produce oils that are unfit for human consumption but can be utilized to make a variety of oleo-chemicals with a wide range of industrial uses. Additionally, oilseeds that produce edible oils are divided into two subclasses. The first type of oilseeds is traditional, which may produce edible oils like canola, soybean, and sunflower for daily use. The other type of oilseeds is non-conventional and produces oils with useful functional qualities, such flaxseed oil, which is often used in relatively tiny amounts. Due to the country's rapid population growth roughly 1.5 million people are added to Egypt's population each year as well as the country's steadily rising edible oil consumption rate per capita in recent years; edible oil consumption has significantly increased. Presently, Egypt uses over 2.5 million tons of edible oils per year, with cottonseed a byproduct of the cotton ginning industry producing approximately 2% of this total amount. In addition to imported soybean oil, less is extracted from local sunflower oilseed. (Atteia shaaban, 2018). The latest data available to the author and which has been kindly supplied by Mist company in Egypt have shown that the quantities of oils imported in Egypt during the period from first of January 2023 to 31 August 2023 were as listed in Table 1.

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Table 1: Quantities of some refined imported oils in Egypt

Imported oil	Quantities per 1000 tons
Sunflower oil	280.859
Soybean oil	167.317
Corn oil	3.636
Palm Olein	326.363

This paper concerns by evaluating and comparing theses oils with respect to their quality as well as for their OS. Since lipid oxidation proceeds very slowly at room temperature, accelerated techniques should be used to determine a product's OS or the autoxidation reaction's induction time more quickly. The Rancimat test has good consistency and is simple to use. Numerous goods' shelf life, as well as the kinetic parameters of antioxidants in oil samples and the antioxidants' ability to suppress lipid peroxidation, have been assessed using the Rancimat method. (Tan *et al.,* 2001; Farhoosh *et al.,* 2008; Chen and Huang, 2016). Kinetic data analysis was made using the previously employed methodology, as described by (Yang and Chiang, 2017; Farhoosh *et al.,* 2008). The oil samples' Induction period (IP) was automatically measured and used as the depicted curves' break point (the point where the two extrapolated halves of the curve overlap).

2. Materials and Methods

2.1. Materials

Fresh refined sunflower, soybean, corn and palm olein oils, all free of artificial antioxidants were provided from Arma Food Industries Company located in10th of Ramadan city, Egypt.

2.2. Methods

2.2.1. Physico-chemical characteristics of the investigated tested oils

The following parameters were measured using the AOAC (2012) method: refractive index, color, acidity (% as oleic acid), and peroxide value. Nelson and Susana's (1995) equation was used to compute the iodine value and saponification value based on the composition of fatty acids.

2.2.2. Determination of fatty acid composition of the investigated tested oils

2.2.2.1. Preparing of fatty acids methyl esters

Using a cold methanolic potassium hydroxide solution, trans-esterification was used to first create the methyl esters of fatty acids in oils, following the procedure outlined in (IOOC, 2001).

2.2.3. Gas liquid chromatographic analysis of fatty acid methyl esters

An Perkin Elmer Auto System XL gas chromatograph fitted with a flame ionization detector (FID) and a fused silica capillary column HP-Wax (30 m x 0.32 mm i.d.) was then used to identify the constituent parts of the produced fatty acid methyl esters. In the GC system, one microliter of the FAME mixture was injected. Ten to one split ratio and an inlet temperature of 240˚C were present. Helium was the carrier gas, flowing at a steady rate of 1 milliliter per minute. After setting the oven temperature to 50˚C for one minute, it was programmed to rise to 220˚C at a rate of 3˚C per minute and remained there for 20 minutes. 450 ml/min of air flow and 45 ml/min of hydrogen flow were used to set the detector to 250˚C. Comparing the relative and absolute retention durations of fatty acid methyl esters to those of genuine pure standards allowed for the identification of fatty acid methyl esters.

2.2.4. Determination of the oxidative stability by Rancimat

Each oil or fat has a level of oxidation resistance that varies depending on saturation, prooxidants, natural or added antioxidants, and history of misuse. Up until this resistance is overcome, oxidation proceeds slowly; beyond that, it quickens and becomes extremely fast. The resistance to oxidation is determined by the duration of time preceding this abrupt increase of oxidation, which is often referred to as the "induction period."

With an air flow rate of 20 L/h, the induction period of the examined oil samples was measured using a professional automated Rancimat Metrohm Co., Switzerland, model 892 at three different temperatures: 90, 100, and 110˚C ± 0.2˚C. Each of the eight reaction jars held three grams of the tested

oils, which were weighed precisely. Based on determining the time (usually referred to as the "induction time") before the maximum rate change of oxidation, the method measures 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 the de-ionized water, as described by Mendez *et al*. (1997).

2.2.5. Kinetic parameters (Ea, Arrh. Factor, ΔH & ΔS) of investigated refined oils oxidation

The kinetic parameters were estimated for investigated oils according to the method described in (Upadhyay& Mishra, 2016).

A kinetic rate constant (k) for lipid oxidation in 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:

$$\ln(k) = \ln(A) - (Ea/RT) \quad \text{.....(1)}$$

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 (ΔH) and entropies (ΔS) of lipid oxidation in investigated oils were calculated using:

$$\ln(k/T) = \ln(kB/h) + (\Delta S/R) - (\Delta H/RT) \quad \text{..... (2)}$$

where kB is Boltzmann constant (1.380×10−23 J/K) and h is Planck's constant (6.63×10−34 J s). The ΔH and ΔS 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).

$$\ln(IP) = a(T) + b \quad \text{.....(3)}$$

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

3. Results and Discussion

The physical and chemical properties of edible oils and fats are varied and have a bearing on consumer acceptability, palatability, and quality assurance. These properties are also linked to the safe and healthy quality standards of these lipids.

The physical and chemical quality assurance criteria of the tested oils including refractive index(RI) , color ,free fatty acid (FFA), peroxide value(PV), iodine value(IV), and saponification number(SN) were determined and the obtained results are recorded in table 2,

Table 2: Physico-chemical characteristics of investigated refined oils

Property		Sunflower oil	Soybean oil	Corn oil	Palm Olein
Refractive index at 25°C		14660±1	14661±1	14630±1	14575±1
Color	**Red**	9±1	1.4±0.1	1.8±0.1	3±1
	Yellow	10±1	16±1	19±1	34±1
Acidity (% as oleic acid)		0.04±0.01	0.02±0.01	0.03±0.01	0.01±0.01
Peroxid value (m equiv O_2/kg oil)		0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Iodine value (g of I/100g oil)		132.05	129.67	118.58	59.15
Saponification value (mg KOH/g of oil)		190.37	194.32	190.37	202.02

*Values are expressed as means of triplicate determination ±SD.

Refractive index: The RI is considered one of the most important physical characteristics, as it is useful for identifying oils and estimating the degree of their unsaturation; the presence of a large

concentration of unsaturated fatty acids in an oil causes its RI to rise, the results in table 2, indicated that the RI of SF, SB, CO and PO oils was found to be 14660, 14661, 14630 and 14575 respectively.
Color: The results in the same Table showed that color was 9, 1.4, 1.8 and 3 in red Lovibond scale, but it was 10, 16, 19 and 34 in yellow scale for the same previous samples, respectively. The color is considered an important visual attribute in the preceptor of product quality.

Acidity%: From the same table, it could be concluded that FFA for all studied oils were ranged from 0.01 to 0.04 (% as oleic acid). The low FFA of all investigated oil samples is considered a good indicator of the success of the neutralization process and suitability of these oils for edible purposes. FFA can be also used as an indicator for the age of the oil (Muhammad *et al*., 2011 and Belewu and Belewu, 2007).

Peroxide value: PV is a measure of the concentrations of peroxides and hydroperoxides generated during the initial phases of lipid oxidation. It is referred to as an indicator of the extent of hydroperoxides. Nonetheless, the lower PV of oils may be a good sign that some compound linked to oxidation and rancidity were not present (Eddy *et al*., 2011). As shown in Table 2, PV for all studied oils was found to be 0.00 (m equiv O_2/kg oil). As viewed the former facts, the investigated oils were characterized with a very good oxidative state when compared with the legislations for the edible oils.
Iodine value: The obtained results Table 2, illustrated that IV of tested oils was found to be 132.05, 129.67, 118.58 &59.15 (g of I/100g oil) for SF, SB, CO and PO oils respectively. IV is a useful metric for determining how unsaturated an oil's fatty acids are. (Xu *et al*., 2007).

Saponification value: As illustrated also in Table 2, SV of all tested oils was 190.37, 194.32, 190.37 and 202.02 (mg KOH/g of oil) for the same mentioned respectively. SV is a measure of the average of molecular weight or, more correctly, it gives an idea about the nature of the chain-length of the fatty acids (Muhammad *et al*., 2011). It is worth to mention that the physical and chemical quality assurance criteria of the tested oils were in conformity with the Egyptian Standards (ES) 49-3, 6, 7 & (ES) no.1706 (2005).

Table 3: Fatty acid composition of investigated refined oils

Fatty acid	Sunflower oil	Soybean oil	Corn oil	Palm Olein
C12:0	---	---	---	0.39
C 14:0	---	---	---	1.03
C 16:0	6.02	11.82	11.31	38.28
C 16:1	---	---	---	---
C 18:0	4.59	3.46	5.17	4.42
C 18:1c	26.04	24.15	25.99	42.74
C 18:1t	---	---	---	---
C 18:2c	62.82	56.06	54.44	12.43
C 18:2t	---	---	---	---
C 18:3	0.33	4.51	0.74	0.33
C 20:0	0.20	---	2.35	0.38
T.S.F.As	10.81	15.28	18.83	44.50
T.U.S.F.As	89.19	84.72	81.17	55.50

The composition of fatty acids determined in SF, SB, CO and PO oils are shown in Table 3. In all samples the main fatty acids were palmitic (16:0), stearic (18:0), oleic (18:1c), linoleic (18:2c) & linolenic (18:3). The analyzed oils exhibited that the total saturated fatty acids(T.S.F.As) were 10.81%, 15.28%, 18.83% &44.50% for the tested SF, SB, CO and PO oils , while a concentration of the total unsaturated fatty acids(T.U.S.F.As) in the same mentioned oils were found to be 89.19%, 84.72%, 81.17% & 55.50 respectively. Also, table 3, revealed that palmitic acid (16:0) was found to be the dominant saturated fatty acid and accounted 6.02%, 11.82%, 11.31% & 38.28 % in all the former tested oils; respectively. With respect to the results, it could be noticed that SF, SB & CO oils are characterized by a high content of Linoleic acid (18:2 ω-6) which was found to be the predominant unsaturated fatty acid in the former three oils and accounted for 62.82%, 56.06%& 54.44% of the total fatty acids; respectively. While, the PO contained (18:2 ω-6) at lower level of 12.43% of the total fatty acids.

Regarding this matter, it is believed that the fatty acids omega-6 and omega-3, which are not produced by mammals and must be obtained through diet, are vital. Furthermore, ω-6 and ω-3 fatty acids are necessary for the body's regular development, growth, and health. (Moreira and Mancini-Filho, 2004). Mean while, Oleic acid (18:1) was the predominant unsaturated fatty acid in PO oil which represented 42.74% of the total fatty acids. Oils that had high oleic acid content are desired because they have a high level of stability (Abdulkarim *et al*., 2005). On the other hand, the SF, SB, CO and PO oils contained trace to small amounts of ($C_{18:3}$) & ($C_{20:0}$), and SBO no containing ($C_{20:0}$). Only PO oil contained much lower amounts of ($C_{12:0}$) & ($C_{14:0}$) which recorded 0.39% and 1.03% of the total fatty acids respectively and disappeared in the other former tested oils.

Table 4: Induction periods of investigated refined oils by Rancimat.

Oil samples	Oxidative Stability by Rancimat		
	Induction period (hrs) at		
	100° C	110° C	120° C
Sunflower oil	10.68	5.67	1.77
Soybean oil	15.63	8.01	2.82
Corn oil	24.11	12.29	4.93
Palm Olein	60.60	18.01	13.75

The induction periods of the studied oils as recorded by the results of Rancimat test are listed in table (4). It is obvious that higher induction period indicate higher oxidation stability of fats and oils. The obtained data indicated that palm olein had highest IP (60.60, 18.0 and 13.75 hrs) which means higher oxidative stability.

It is important to note that the oxidation of edible oils causes complex and varied changes that affect their functional quality and yield a variety of breakdown products. (Lalas, 1998).

Furthermore, the oxidation stability of fats and oils gives us insight into potential uses for culinary or industrial goods, with the exception of their shelf life and susceptibility to oxidative rancidity during processing and storage. (Anwar *et al*., 2003). High unsaturation oil and fats are more vulnerable to radical assault as an initial stage of oxidation. In general, fatty acids with one or two double bonds are less vulnerable to oxidation than polyunsaturated fatty acids (C18:2, linoleic acid; C18:3, linolenic acid). (Knothe &Dunn, 2003). As can be seen from the above result in Table (4), Rancimat was used to measure the oxidation rates of the oil samples under investigation at three distinct temperatures: 100, 110, and 120°C. According to the data gathered, PO oil had the highest IP (60.60, 18.01& 13.75 hours, Rancimat; 20L/hrs, 100, 110, and 120°C, respectively); this is an attribute of oils' OS. Thereupon, the PO oil was exhibited a higher good resistance to the oxidation rancidity than those for CO oil (24.11, 12.29& 4.93 hrs), followed by SB oil (15.63, 8.01& 2.82 hrs) and then SF oil (10.68, 5.67& 1.77 hrs)(Rancimat; 20L/hrs, 100,110& 120° C). A high good OS of PO oil was due mainly to a significant higher level of $C_{16:0}$ and $C_{18:1c}$ (38.28% &42.74%), which is less prone to the oxidation than polyenoics. This has been clearly demonstrated in the other studied oils (SF, SB & CO), which had a lower OS and high content of Linoleic acid (18:2 ω-6).

Table 5: Shelf-life (IP_{25}) prediction of investigated refined oils

Oils	Shelf-life (IP_{25})	$\ln(IP) = a(T) + b$	
	Days	Months	Years
Sunflower oil	529.51	17.65	1.47
Soybean oil	508.74	16.96	1.41
Corn oil	445.83	14.86	1.24
Palm Olein	589.90	19.66	1.64

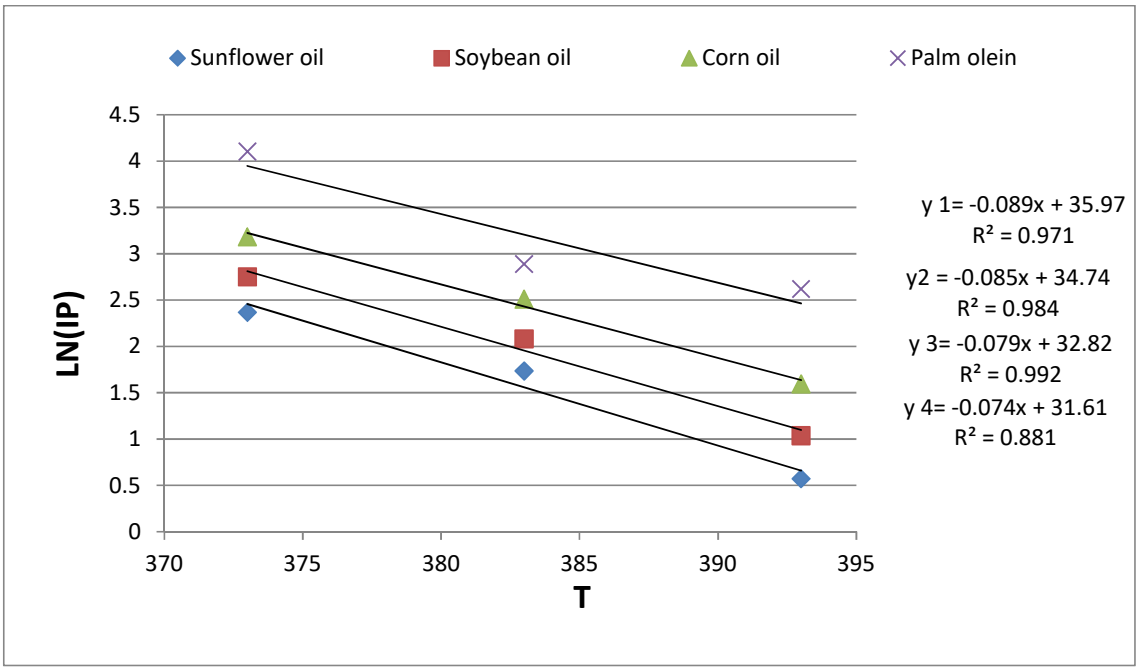

Fig. 1: Semi-logarithmic relationship between ln(IP) and temperature values (T) for Shelf-life (IP25) prediction of investigated oil samples.

As indicated in the obtained data Table 5 and Fig.1, the Shelf-life hours, days and months (predication index IP_{25}) of the tested oils was calculated by plotting the natural logarithm of IP vs. absolute T (K) using Eq. ($\ln(IP) = a(T) + b$). The higher good resistance to the oxidative rancidity (IP_{25}) were exhibited for PO oil 19.66 months than those the other tested oils which recorded 17.65, 16.96, 14.86 months for SF, SB and CO oils respectively. Thereupon, the Shelf-life periods at ambient temperature of former tested oils were decreased in order: PO > SF> SB> CO.

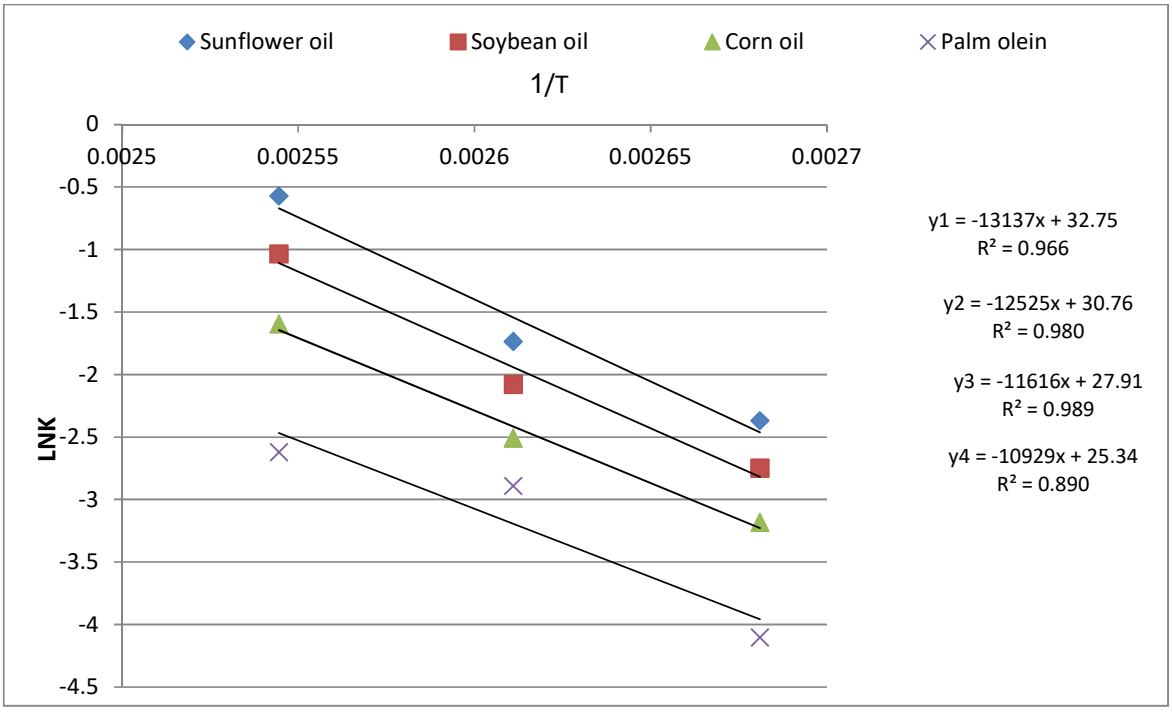

Fig. 2: Semi-logarithmic relationship between ln(k) and temperature values (1/T) for lipid oxidation of investigated oil samples.

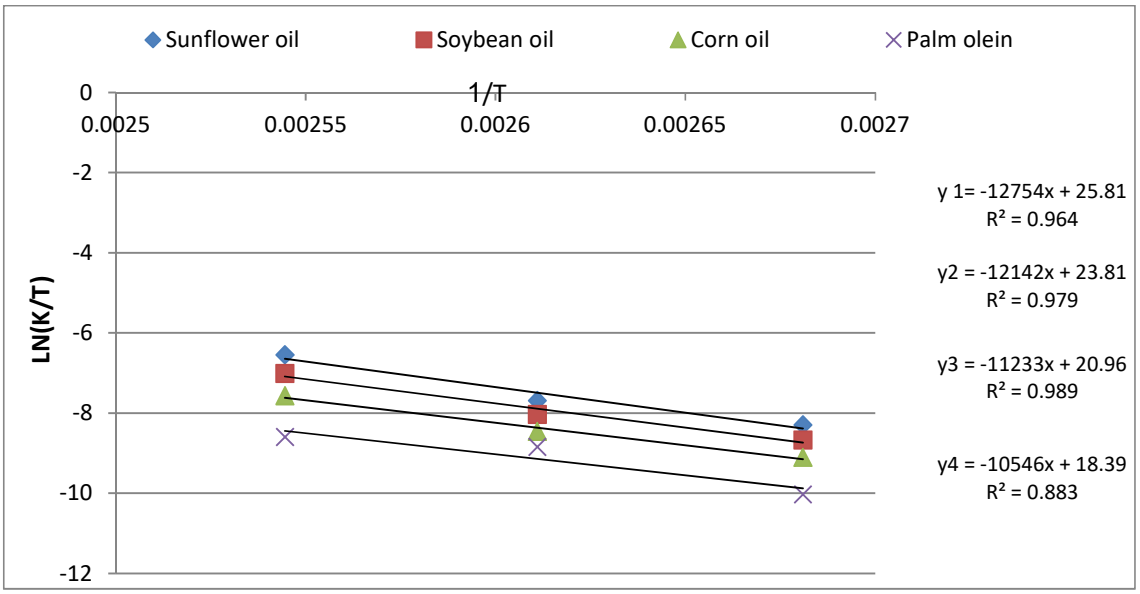

Fig. 3: Semi-logarithmic relationship between ln(k/T) and temperature values (1/T) for lipid oxidation of investigated oil samples.

The Rancimat test findings can be used to estimate a number of kinetic parameters related to the oxidation process. They can be used to distinguish between different oils and to find similarities and differences among them. When it comes to anticipating the oxidative stability of oils after heat treatment, storage, and distribution, these criteria could be extremely helpful (Tan *et al.,* 2001). For each of the examined oils, correlations between temperature and the logarithm induction time τ Rancimat as well as the opposite of temperature were found (Figs. 2 and 3). These correlations could be found using equations 1 and 2, and they had a linear nature with correlation coefficients of R2 of 0.966 for the SF, 0.977 for the SB, 0.989 for the CO, and 0.890 for the PO oil samples.

Table 6: Kinetic parameters (Ea, Arrh.Factor, ΔH & ΔS) of investigated refined oils oxidation.

Oils	Ea(KJ/mol)	Arrh.FactorA,h^{-1}	ΔH(KJ/mol)	ΔS(J/mol k)
Sunflower oil	109.22	$1.67X10^{14}$	106.04	17.04
Soybean oil	104.13	$2.29X10^{13}$	100.95	0.42
Corn oil	96.58	$1.32X10^{12}$	93.39	-23.28
Palm Olein	90.86	$1.01X10^{11}$	87.68	-44.65

As evident in Table (6), the following kinetic parameters (Ea, Arrh.Factor, ΔH & ΔS) were calculated for SF, SB, CO and PO oil samples. The Ea value is significant for the characteristics of oils since a decrease in Ea values indicates a slower rate of lipid oxidation and, consequently, a delay in the first oxidation reaction because of the bond scission that results in the formation of primary oxidation products (Adhvaryu *et al*., 2000). The Ea values of the former tested oils were 109.22, 104.13, 96.58 & 90.86 kJ/mol for SF, SB, CO and PO oils, respectively. Thereupon, the OS of the investigated oil samples was in order: PO>CO>SB>SF. Also, the results in the same table indicated that the Arrh. Factor of the former tested samples was being $1.67X10^{14}$, $2.29X10^{13}$, $1.32X10^{12}$& $1.01X10^{11}$ for SF, SB, CO and PO oils, respectively. In addition, the results indicated also, that even simple changes in Ea led to significant changes in Arrh.Factor. Based on the active complex theory and linear regression results, ΔH and ΔS were computed and entered into Tables (6). The ΔH value determined for the analyzed refined SF, SB, CO and PO oil samples, which ranged from 87.68 kJ/mol for PO oil to 106.04 kJ/mol for SF oil. In turn, the ΔS value ranged from -44.65 to 17.04 J/mol K for the same refined oils .The reduction in ΔH & ΔS values indicated to a slower rate of lipid oxidation. Moreover, the negative ΔS values indicate a slower rate of lipid oxidation and a decreased likelihood of the development of an

active complex (Farhoosh *et al*., 2008). The aforementioned assertion is accurate for the refined oil samples under investigation, according to the findings of our study.

4. Conclusion

In conclusion, the Rancimat test was used to assess the oxidative stability of all refined oils under investigation at three different temperatures being 100,110 and 120°C and the result indicated that the palm olein (PO) oil is characterized by higher values of oxidation rate constant followed by corn (CO) then soybean (SB) and sunflower (SF) oils. In turn, the thermo kinetics properties (Ea, Arrh.Factor, ΔH and ΔS) were estimated for all tested oils and the obtained data showed a reduction in values of Ea, ΔH and ΔS for PO oil followed by CO then SB and SF oils. This reduction in kinetics parameters indicated to a slower rate of lipid oxidation and hence, delayed of the initial oxidation.

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6. Conflicts of interest
No competing interests are disclosed by the authors.

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