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The stability of vegetable oils (sunflower, rapeseed and palm) sold on the Moroccan market at high temperature

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Abstract

In this work, the physico-chemical parameters of sunflower, rapeseed and palm oils were measured after every 6 hours. These oils were treated in the same thermo-oxidative state for 30 hours at 180°C. The evaluation of oxidation was studied by measuring the peroxide value, the absorption at E270, free fatty acid, iodine value, fatty acids composition and polar compounds. The results showed that palm oil was comparatively more stable at high temperatures than other oils. The levels of polar compounds were remains low in the palm oil, even after 30 hours of heating.

Key words: vegetable oil, stability, heat treatment, polar compound

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1. Introduction

Edible oils are from vegetable origin obtained by extraction of oilseeds (soybeans, rapeseed, sunflower, argan, peanuts, etc) or oleaginous fruits like coconut, olive and palm, or from animal fat like the pork fat (lard) or cattle (tallow), but essentially from some products deep sea fishing (cod, whale). Since the turn of century, vegetable oils have gradually replaced animal oils as main source of food fat. Vegetable oils consist of triglycerides between 95 and 99% [1]. They can also contain soluble vitamins (A, D, E and K), phytosterols, natural pigments and phospholipids from 1 to 5% [1]. The constituent fatty acid (FA) of triglycerides differs from each other by the length of their carbon chain and the number of double bonds [2]. Vegetable oils play an essential role in the diet [3, 4], they ensure nutritional function: they contribute to the energy supply, are essential sources of fatty acids (linoleic acid C18: 2, the precursor of the family of omega-6 and α-linolenic acid C18: 3, a precursor of omega-3). These two fatty acids are considered essential because they cannot be synthesized by humans, and are essential for the proper functioning of the human body, growth and physiological functions, on the other hand they contribute to the prevention of certain diseases related to eating habits (cardiovascular diseases, diabetes, obesity, cancer), so therefore they must be supplied by food [5,6]. However, these unsaturated fatty acids are susceptible to oxidation [7]. Lipid oxidation has a negative impact on the functionality of raw materials, sensory and nutritional quality of food, and causes economic losses [8]. The most noticeable result of lipid oxidation is the appearance of an unpleasant flavor often referred to rancid, which modifies the sensory characteristics of the food, so its assessment by the consumer [9,10, 11,12].

Lipid oxidation also led to a change in color and sometimes texture, as well as the loss of essential nutrients and micronutrients [11]. Finally, lipid oxidation can lead to the formation of potentially toxic oxidation products (oxycholesterol, malonaldehyde, endoperoxides, acrolein, polymeric peroxides) [13, 14]. The oxidation of unsaturated fatty acids phenomenon has been studied for more than half a century [15-20]. The main mechanisms involved are described in the literature and the kinetics and factors of variation generally well known. The report of Lipid oxidation is an auto-catalytic reaction [16]. It is a sequence of radical reactions drop broadly into three stages: initiation, propagation and termination [20]. The oxidation of unsaturated fatty acids may result from three reaction pathways, depending on the environment and initiators: (i)

self-catalyzed oxidation temperature, metal ions and free radicals, (ii) oxidation initiated by lipoxygenase enzyme and (iii) photo-oxidation, essentially under UV and in the presence of sensitizers, catalyzed by singlet oxygen [19, 21, 22]. These three reaction pathways lead to the formation of a family of compounds, hydroperoxides (LOOH) [20-22]. Factors that influence lipid oxidation are numerous. It is part of intrinsic factors such as the composition of unsaturated fatty acids (concentration and number of unsaturations), the water activity, the presence of pro-oxidants (metal ions, hemes, enzymes) or natural antioxidants (tocopherols, carotenoids, ...), and secondly of the major environmental factors which are the temperature, light and oxygen partial pressure [2, 7, 9, 12, 22]. An increase in temperature favors the oxidation of lipids. Indeed lipid oxidation is more rapid as the temperature is higher either for industrial or thermal treatments, domestic use (cooking, frying) or during their storage [7, 12, 23-25]. The aim of this study is to comparing the chemical changes that occur after exposure to prolonged heating conditions: 180 °C for 30 hours. We chose for this study, sunflower, palm and rapeseed oil, as these oils are widely used all over the world as frying medium [3].

2. Material and Methods

2.1. Chemicals

All reagents used were of analytical or HPLC grade. 2,2,4-Trimethylpentane, heptane and Hexane used for chromatography, and cyclohexane, acetic acid, methanol, ethanol, Wijs, reagent petroleum and diethyl ether used respectively for extinction coefficient determination, peroxide value, free fatty acid, peroxide value and polar compounds were purchased from Professional Labo (Casablanca, Morocco)

2.2. Sample preparation

Sunflower, rapeseed and palm oils were obtained from Lesieur Cristal, Roches Noires, Morocco.

2.3. Physicochemical parameters

Determination of physicochemical parameters (free fatty acid, peroxide value, light absorption K270 and iodine value), were carried out according to the analytical methods described by Regulation EEC/ 2568/91 and EEC/ 1429/92 of European Union Commission [26]. Free fatty acid, given as percentage of oleic acid, was determined by titration of a solution of oil dissolved in EtOH/Et2O (1:1) with 0.1 M KOH in EtOH. To determine the peroxide value, expressed as milli-equivalents of active oxygen per kilogram of oil (meq/kg), a mixture of oil and iso-octane – acetic acid was left to react with a solution of KI in the darkness; the free iodine was then titrated with a sodium thiosulfate solution. K270 extinction coefficient was determined from absorption at 270 nm, with a UV spectrophotometer (CARY 100 Varian UV spectrometer), using pure cyclohexane as a blank.

2.4. Fatty acids composition

Fatty acid composition was determined following regulation EEC/2568/91 [26] as previously described [27]. Into 5 mL screw top test tubes, 0.60 g of oil and 4 ml of iso-octane were introduced, followed by 0.20 mL of 2N methanolic KOH solution was added. Tubes were tightened with a screw cap provided with a PTEF joint, and then vigorously shaken. The upper layer was separated and fatty acid methyl esters (FAMEs) were extracted with hexane. (FAMEs) were analyzed by gas chromatography using a Varian CP-3800 (Varian Inc.) chromatograph equipped with a FID. A split injector was used and the injected volume was 1 µl. The column used was a CP-Wax 52CB column (30 m · 0.25 mm i.d.; Varian Inc., Middelburg, The Netherlands). The carrier gas was helium and the total gas flow rate was 1 mL/min. The initial and final column temperature was 170 and 230 °C, respectively, and the temperature was increased by steps of 4 °C/min. The injector and detector temperature was 230 °C. Data were processed using a Varian Star Workstation v 6.30 (Varian Inc., Walnut Creek, CA, USA). Results were expressed as the relative percentage of each individual FA present in the sample.

2.5. Oxidative stability of oils

Oil oxidative stability was evaluated by the Rancimat method [27]. Stability was expressed as the oxidative induction period (IP, hrs) measured at 110 °C on a Rancimat 743 (Metrohm Co, Basel) apparatus using 3 g of oil sample with an air flow of 20 L/hr. Volatile oxidation products were stripped from the oil and dissolved in cold water, whose conductivity increased progressively. The time taken to reach a level of conductivity was measured

2.6. Polar compounds

Total polar compounds were determined by adapting the method of Waltking and Wessels [28]. To a 50 mL volumetric flask, 2.5 g of oil was introduced and the volume adjusted to 50 mL using a solution of light petroleum and diethyl ether (87:13, v/v). Twenty mL of this solution was transferred to a chromatography column (30 X 1.8 cm i.d.) containing 20 g of silica deactivated with distilled water and suspended in a n-hexane/diethyl ether (87:13) mixture. Elution was first achieved using 250 mL of this solvent and the eluate was collected. Then, elution was repeated with 150 mL of the same solvent and the eluate collected in a separate flask. Both fractions were determined gravimetrically once the solvent had been evaporated

2.7. Statistical analysis

Values reported in tables are the means ± SD of 3 replications. The significance level was set at P=95% and (*alpha* = 0.05). Separation of means was performed by Tukey's test at the 0.05 significance level.

3. Results and Discussion

3.1. The initial oxidative stability by Rancimat

The oxidative stability index of oils (induction period time) was given in Table 1. The Rancimat induction time at 110 °C varied from 4.5 h to 15.5 h in the specialty oils samples. among tree tested oils, the rapeseed oil was the least heat stable oil on the other hand the palm olein oil was the most stable (15.5h) followed by sunflower oil (5.5h). the high stability of palm olein oil is most likely due to its fatty acids composition which contained nearly 40% of saturated fatty acids (SFA) and this can be a reason for the comparatively lower Rancimat value of rapeseed and sunflower oil even they had close levels of total PUFA and SFA (see table 2). Miraliakbari and shahid; 2008 [29] noted that walnut oil as the least stable and most unsaturated

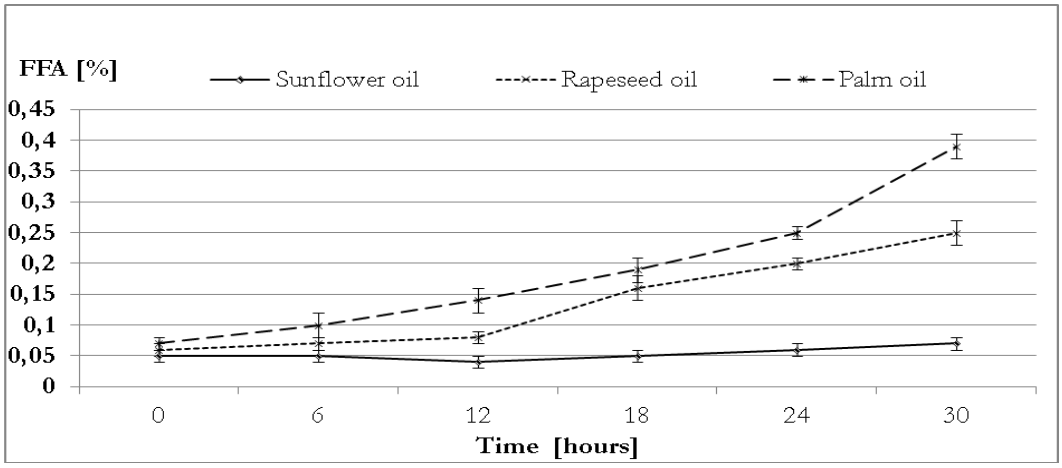

Fig. 1. Evolution of free fatty acid content depending on the time of heating at 180 ° C.

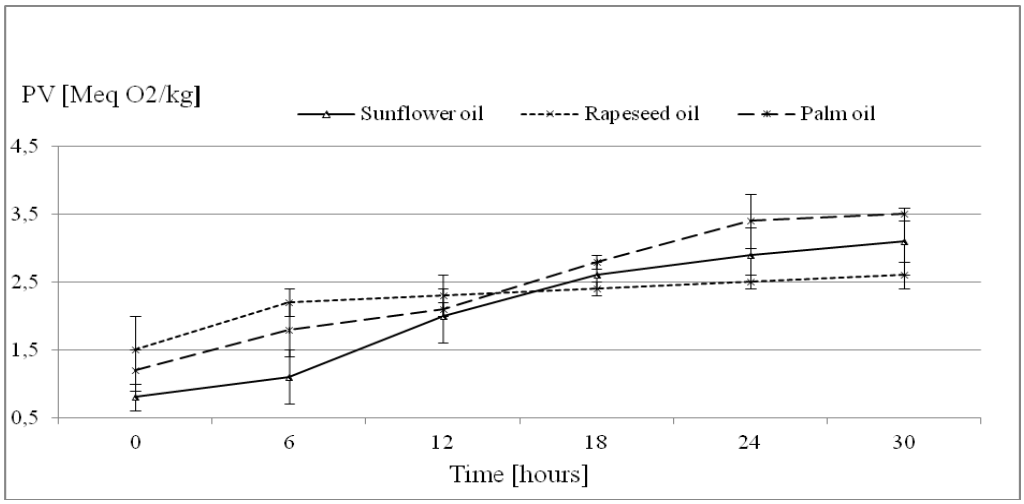

Fig. 2. Evolution of the peroxide value depending on the time of heating at 180 ° C.

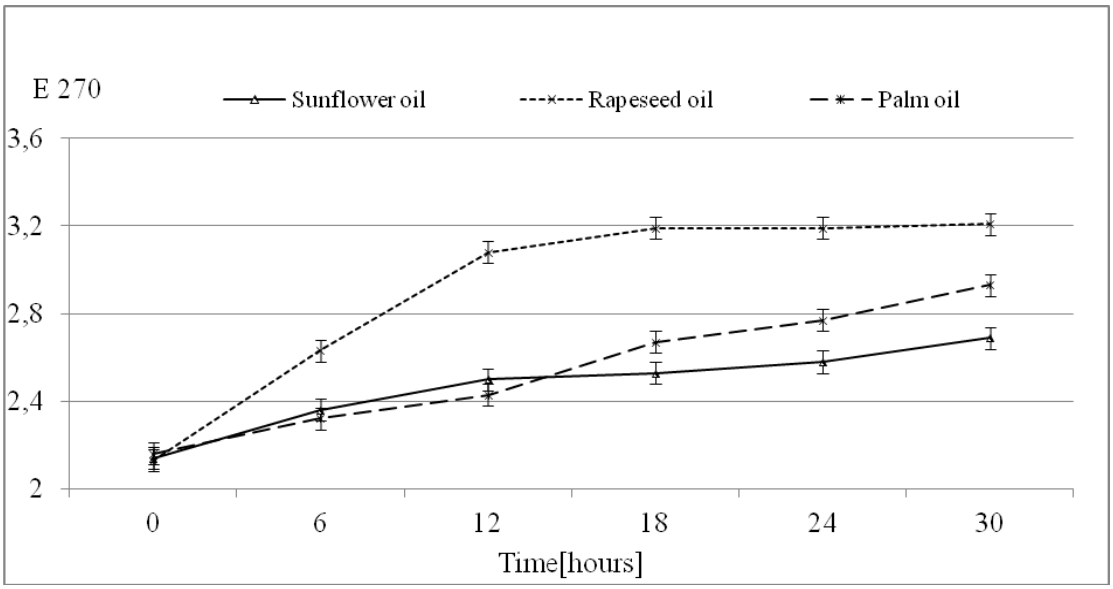

Fig. 3. Evolution of E 270 depending on the time of heating at 180 ° C.

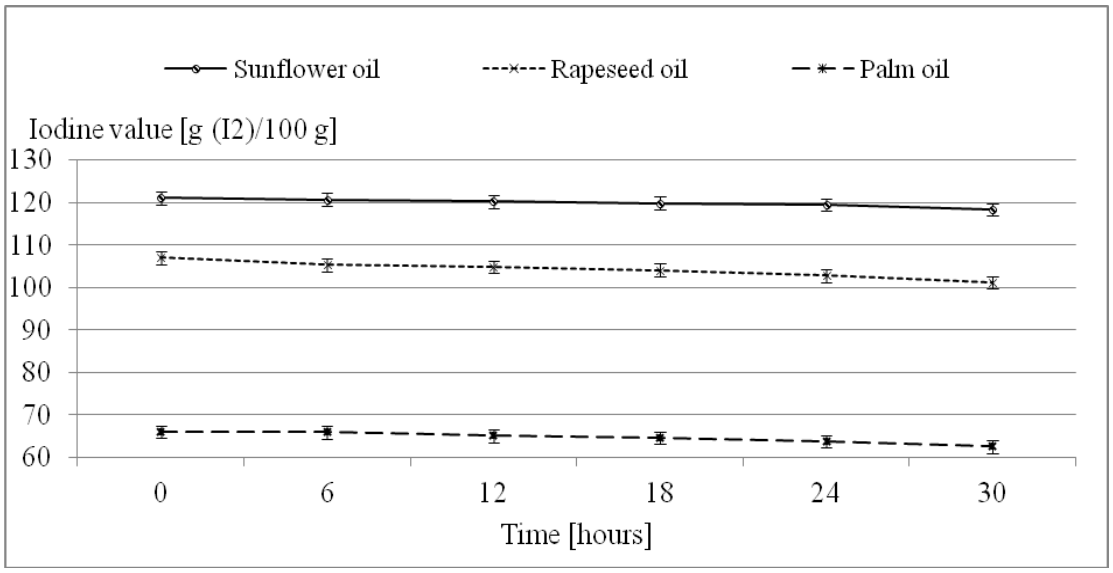

Fig. 4. Evolution of iodine value depending on the time of heating at 180 ° C

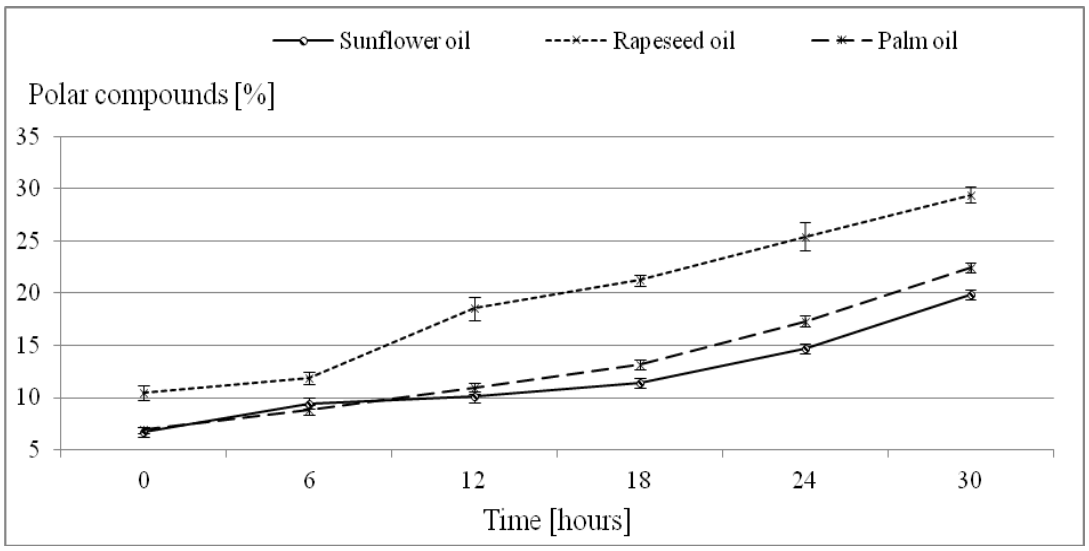

Fig. 5. Evolution of polar compounds depending on the time of heating at 180 ° C.

Table 1. Rancimat at 110 ° C of the oil palm olein, sunflower and rapeseed

	Palm olein	Sunflower	Rapeseed
Rancimat [h]	15.5 ± 1.5	5.5 ± 0.5	4.5 ± 0.5

Mean ± SD: Standard deviation values are expressed as mean of samples analyzed in triplicates.

Table 2. Degradation of fatty acids according to the heating period.

(%) Fatty acids	Sunflower		Palm olein		Rapeseed	
	0h	30h	0h	30h	0h	30h
Palmitic acid	6.5±0.1	6.7±0.1	35.5±0.1	36.3±0.1	4.9±0.1	5.5±0.1
Stearic acid	3.7±0.1	3.6±0.1	4.9±0.1	5.3±0.1	1.5±0.1	1.6±0.1
Oleic acid	34.8±0.1	35.1±0.1	42.3±0.1	43.5±0.1	59.9±0.1	62.8±0.1
Linoleic acid	53.6±0.1	52.9±0.1	14.5±0.1	12.2±0.1	19.8±0.1	18.1±0.1
α-Linoleic acid	0.1±0.1	-------	0.9±0.1	0.5±0.1	10.4±0.1	8.1±0.1
SFA	10.2±0.1	10.3±0.1	40.4±0.1	41.6±0.1	6.4±0.1	7.1±0.1
PUFA	88.5±0.1	88±0.1	57.7±0.1	56.2±0.1	90.1±0.1	89±0.1

SFA: Saturated fatty acids. PUFA: Polyinsaturated fatty acids, Mean ± SD: Standard deviation values are expressed as mean of samples analyzed in triplicates

while almond and hazelnut exhibit intermediate stability in a study on the oxidative stability of tree nut oils.

3.2. Change in quality indices during heating to 180 ° C

3.2.1. Free fatty acid

Changes of acidity or free fatty acids (FFA) were shown in the figure 4. Results showed that FFA increased significantly to reach high levels at the end of induction time respectively for palm olein (vary from 0.07 to 0.39%) and rapeseed oil (vary from 0.06 to 0.25%), while we didn't note any variation in FFA for sunflower oil which remains constant during heating time. The presence and increase of FFA has been shown to catalyse both oxidation and hydrolysis of triacylglycerols and consequently they contribute to the decrease of the smoke point due to their partial volatilisation [30]. The invariability of FFA for sunflower oil might due to its high thermal stability confirmed by some studies [31], normally the palm olein should display same results as sunflower in point of its high stability. its seems that each oil perform a specific behaviour towards heating treatment, which might explain the observed data and should be confirmed by establishing many correlation between different parameters such as Rancimat index, FFA levels, oxidative stability and fatty acid composition.

3.2.2. The peroxide value

The peroxide value (PV) of different oils was shown in the figure 2. The PV of rapeseed oil increased rapidly during the first 6 hours, it reached (2.25 Meq O_2/kg oil) comparing to the beginning time (1.25 Meq O_2/kg oil), this value remains relatively constant in the following hours. In contrast, the palm olein and sunflower oil showed a slight variation after 6 hours comparing to the rapeseed oil. However, they increased significantly at the end of the induction time (30h). In our study, we showed that PV of rapeseed oil did not vary after 6 hours depending on the heating time which normally should increases intensively due to the oxidative activation. Consequently, in this case we cannot consider this index as an appropriate indicator to evaluate the hydroperoxides degradation at high temperature; in fact it can be used as an index of instability [32, 33]. for sunflower and palm olein oils, the PV was increased successively from (0.8 Meq O2/kg oil) to (3.1 Meq O2/kg oil) at 30h and from (1.2 Meq O2/kg oil) to (3.5 Meq O2/kg oil) at the end (30h), this might be due to high amounts of polyunsaturated fatty acids compared to the rapeseed oil. Indeed, the observed results are in accordance with many recent studies investigating the production peroxide of oils using heat treatment [34, 35]. At this level, even we noted a variation of peroxide oils related to heating time we cannot firmly establish a clear conclusion about the peroxide index of the using oils and their heating behavior [35].

3.2.3. E 270 study

Carbonyl compounds (aldehydes and ketones) are the most abundant secondary oxidation products formed in edible oils. Their formation is known to be accelerated by elevated temperature and metal traces [11; 22]. UV absorption at E 270 nm (K270) is one of the markers used to follow secondary oxidation formation [36]. These values increased progressively through the oxidation period at (180 °C, 30h). The results showed that E270 value varied from 2.13 to 2.69 and 2.93 successively for sunflower and palm oil. The high absorption E270 was observed in rapeseed oil reached 3.21 at the end of heating time (30h) noted by an increase of 33.64% comparing to the beginning time. The elevated amount of K270 in rapeseed oil might be resulting of high concentration of conjugated trienes as a

secondary oxidation product due to the oxidation process [37]. Related to our results, rapeseed oil has a great value of peroxide (PV), this indices is highly correlated with K270 [21] which is characterized by a formation of secondary oxidation product especially hydroperoxides [38]. Other studies were consistent with ours, confirming the correlation between PV and E270 testing at 120 °C [35]. Thus, the rapeseed oil stability varied significantly during heating time comparing with sunflower and palm oil.

3.2.4. The iodine value

The iodine value (IV) is a measure of the unsaturation of fatty acids. It is characteristic for a given fat and can be used for its identification. During the heat treatment a slight decrease of iodine value was observed for all oils after 30h at 180 °C of heating. they respectively drop about 2 units for palm olein pass from 66 mg I_2/100g oil to 64 mg I_2/100g oil, 3 units for sunflower oil (120 mg I_2/100g oil to 117 mg I_2/100g oil) and 6 units for rapeseed oil (107 mg I_2/100g oil to 101 mg I_2/100g oil), this decrease can be attributed to the destruction of double bonds by oxidation, scission and polymerisation [39]. Analogous to results obtained from total polar material (TPM). The rapeseed oil had the fastest loss of unsaturation of oils heated at 180 °C compared to palm olein and sunflower oil, thus the IV dropped to a low value when oils with polar compounds have more than 25%, this data match well with our results.

3.2.5. The fatty acids composition

Oil fatty acid (FA) composition is an essential indicator of its nutritional value [40] Our results showed that all tested oils contained large amount of unsaturated fatty acids; the most abundant was the mono unsaturated oleic acid (C18:1) represented successively in palm olein and rapeseed oil by 42% and 60% and the bi-unsaturated linoleic acid (C18:2) in sunflower oil by 53% (Table 2). The temperature treatment at high levels (180 °C) was without changes in fatty acid content in sunflower oil; whereas a slight decrease about 15% and 11% was observed respectively in palm olein and rapeseed oil after 30 hours. thus; it seems that the lower loss of total fatty acids from the latest two oils compared to sunflower oil belong to their lower content of linoleic acid which is ready oxidized and also to their high amount of oleic acid which is less ready to oxidized [41]. In our study the onset temperature of thermal degradation (180 °C) of rapeseed and palm oil decreased with the increase in frying time causing by the decrease in unsaturation due to heating time; thus; it means that the thermal stability of studied oils is dependent on the composition of fatty acids and the loss of stability is accelerated by an increase in process time; which is responsible for changes in the physicochemical and organoleptic characteristics of oils. In contrast, the sunflower oil which is abundant in bi-unsaturated fatty acids put up its low resistant to the thermal decomposition which cannot be consider as a good frying and cooking oil [42].

3.2.6. Polar compounds during heating

The amount of polar compound in the oil samples was shown in figure 5. our results showed that sunflower oil and palm olein had the lowest (~6.5%) and the rapeseed oil has the highest (11%) polar compounds at the beginning of the test, these amounts reached (25.5%) for rapeseed oil after 24 hours. as a basis for assessment of the end point of frying oil, we chose 25% (TPM) which is the regulatory limit in many European countries [42, 43]. according to this, the rapeseed oil surpasses the limit at the end of the experience (30 h) while the other oils remain under limit. generally, oils with high levels of unsaturated fatty acids produced more polar compounds compared to the saturated ones, our results showed that rapeseed oil was the most unsaturated of all oils.

4. Conclusion

Referring to the study of the thermal stability of vegetable oils, our results indicated that the palm olein oil has an excellent profile in terms of stability at high temperature. This stability is due to its acid profile in less affluent to the most sensitive to oxidation unsaturated fatty acids. These results position the palm olein compared to other oils for use in frying. Hence, further investigations will be necessary in order to draw conclusions about how long oils can be heated before the deterioration increases to such a level that it is no longer acceptable for human consumption.

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