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Comparative Study of Argan and other Edible Oils Stability under Accelerated Oxidation

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Abstract

Objectives: The study aimed at assessing the oxidative stability of the commercial Argan oil compared with those of high culinary use in Morocco, namely olive, rapeseed and sunflower oils. **Methods/Analysis:** The oils were subjected to two parallel accelerated oxidation tests, Swift and the oven test at 65 °C in air during 10 weeks. The evolution of oxidation throughout this period was followed periodically by simultaneously measuring the Peroxide Value (PV), the Refractive Index (RI) and absorption at 232 and 270 nm. **Findings:** Swift test showed that Argan oil was the most stable with a period of more than 32 hours to reach a peroxide value of 100 meqO_2/kg at 100°C. The storage at 60°C also revealed that Argan oil was more stable than the other oils. These results showed that the oxidative degradation depended on the initial chemical composition of oils, especially the content of polyunsaturated fatty acids and antioxidants. **Improvement:** The high stability of Argan oil was due to its particular fatty acid composition and its richness in tocopherols and phospholipids that act as antioxidants. The stability of Argan oil may also be enhanced by the Maillard reaction products resulting from the almonds roasting during the extraction.

Keywords: Accelerated Oxidation, Argan Oil, Oxidative Stability, Phospholipids

1. Introduction

Vegetable oils play an important role in the human diet. They are a source of energy, fat-soluble vitamins and essential fatty acids. The oils of sunflower, soybean, rapeseed and olive considered as conventional, are widely used in the culinary field. However, exposure to high temperatures during cooking in the presence of oxygen, leads to a series of reactions such as thermal oxidation, isomerisation, polymerization and hydrolysis of unsaturated fatty acids[1-3]. The derivatives of these reactions are responsible for losses in nutritional characteristics of the oils, and for formation of toxic substances[4]. However, vegetable oils do not have the same oxidation stability due to their different chemical composition particularly their contents of polyunsaturated fatty acids and antioxidants[5-7]. The rate of unsaturated fatty acids oxidation increases with the number of double bonds. Thus, linolenic acid (n = 3) is more susceptible to the oxidation than linoleic (n = 2) and oleic acids (n = 1)[8]. Antioxidants, such as tocopherols, polyphenols and phospholipids, increase the stability of vegetable oils by limiting the action of free radicals on the polyunsaturated fatty acids. However, the content of antioxidants varies depending on various factors such as the extraction mode, the refining processes and the storage conditions[9-10].

Among dietary fats produced in Morocco for local consumption or for export to other countries, Argan oil, extracted from the seeds of *Argania spinosa* L. (Sapotacae), has a privileged position because of its organoleptic and nutritional properties. In this regard, several studies have shown its hypolipideminat, hypocholesterolemiant, anticancer and antidiabetic effects[11]. To preserve these properties, Argan oil is extracted manually or by press following roasting almonds contained in *A. Spinosa* kernels. The oxidative stability studies reported that Argan oil keeps better when protected from light and oxygen[12,13]. However, the marketing of Argan oil is

essentially based on exposure to supermarket shelves in high illumination. The aim of this study was to assess the oxidative stability of Argan oil initially exposed to high illumination, in comparison to other oils sold in Morocco, namely sunflower, rapeseed and olive oils. To do this, the four oils were subjected to accelerated oxidation by using the oven and Swift tests. The oxidation was estimated by simultaneous measurement of the Peroxide Value (PV), the Refractive Index (RI) and the absorption at 232 and 270 nm (K232 and K270).

2. Material and Methods

2.1 Oils

Edible oils were purchased at a local supermarket. They were as follows: extra virgin Argan oil (Dargan), extra virgin olive oil (Jawhara), refined rapeseed oil (Golden Seed, Lesieur) and refined sunflower oil (Huilor). These oils were used in their recommended consumption periods. Their content in fatty acids and tocopherols, mentioned on packaging, is presented in the Table 1.

2.2 Oxidation Parameters

The analyses were performed on all the oils at the same time. The day of purchase was considered as the beginning of the experiment and corresponded to the initial state.

The Acid Value (AV) and the PV were determinate according to IUPAC 2.201[14] and IUPAC 2.501[15] respectively.

The RI was measured at 20 °C using Abbe type refractomter (Atago Co.). The absorbance at 233 and 270 nm was determinate using a Varian DMS 80 spectrophotometer after the oils dilution in cyclohexane (V:V).

2.3 Determination of Phospholipids Content

The phospholipids content was estimated by measuring phosphorous according to AOCS recommended method Ca 12- 55[16]. The method is based on ashing the oil sample in the presence of zinc oxide. The phosphorus is then determinate by a spectrophotometric measurement as a blue phosphomolybdic acid complex (Varian DMS 80 spectrophotometer). The phospholipids content is obtained by multiplying the phosphorus content by 25.

2.4 Evaluation of the Oxidative Stability

The oxidative stability of the oil was studied using storage at 65 ° C with aeration for 10 weeks. It was completed by Swift test that measures the time required for an oil sample under carefully controlled conditions (100 °C in air) to reach a PV of 100 meqO_2/kg of oil.

2.4.1 Storage at 65 °C

The four oils were distributed into beakers (100 ml capacity) at 60 g oil per beaker. The recipients were then kept open in the dark in an oven at 65 °C under continuous aeration for a period of 10 weeks. Oils samples were taken every week for the determination of oxidation parameters namely the PV, the RI and the absorption at 232 and 270 nm.

2.4.2 Swift Test

The technique according to AFNOR was used[17]. Briefly, oils samples (20 ml) were put into test tubes and placed for 5 min in boiling water and then in water bath at a temperature of 100 °C under an air flow of 140 ml per min. After a fixed period, the oxidation was stopped by

Table 1. Initial fatty acid and tocopherols contents in the used oils

Compounds	Oil			
	Argan	Olive	Sun flower	Rapeseed
Saturated fatty acids (%)	18	16,8	11,3	7,6
Monounsaturated fatty acids (%)	45	71	27	63
Polyunsaturated fatty acids (%)	37	12,7	61,3	30
ω 3 fatty acids (%)	0,2	0,5	0,15	9
ω 6 fatty acids (%)	36,8	12,2	61,15	21
Total Tocopherols (mg/kg)	450	100-200	500-800	280-450

rapid cooling the tubes in ice water. This operation is repeated with a longer duration for obtaining a second peroxide value. The two values (comprised between 75 and 175 meqO_2/kg) are used to graphically determine the oxidation duration corresponding to a PV of 100 meqO_2/kg. The value of this time is considered a marker of resistance to oxidation. The test is repeated three times for each extraction method.

2.5 Statistical Analysis

The values represent the mean of three replicates. The results were subjected to ANOVA single factor. The mean values were compared by an averaging multiple comparison test using the least significant difference test (LSD, P = 0.05).

3. Results and Discussion

The initial physico-chemical characteristics of the studied oils are shown in Table 2. The PV varied significantly between oils. It was lowest in Argan (0.63 meqO_2/kg) and highest in olive oil (16.33 meqO_2/kg). Intermediate values were recorded for sunflower and rapeseed oils (4.86 and 7.64 meqO_2/kg respectively). The PV assesses the initial oxidation state of oils. Here, the olive oil had the most advanced oxidation state. Indeed, chlorophyll pigments contained in this oil have a pro-oxidant due to their photo-sensitizer power that produces singlet oxygen. Because of its instability, the singlet oxygen reacts with the unsaturated fatty acids resulting in the formation of hydroperoxides[18]. However, the PV obtained for the four oils remained acceptable and conform to the quality criteria set by the food standards[19].

The initial acidity values were also significantly different (Table 2). Thus, the degree of acidity was low in refined oils (0.05 and 0.11 % for rapeseed and sunflower oils respectively). It was higher in unrefined oils (0.45 and 1.02 % for Argan and olive oils respectively). This difference is explained by the fact that the industrial refining removes free fatty acids by neutralization with alkaline substances. In the case of virgin oils, the values recorded were consistent with those of Moroccan standard[20] and the International Olive Oil Council[21] corresponding to virgin and extra virgin quality respectively for olive and Argan oils.

The values of the RI were significantly different: 1.469 (olive), 1.470 (Argan), 1.473 (rapeseed) and 1.474 (sunflower). These differences are related to the composition and physico-chemical characteristics of each type of vegetable oil, particularly the length of the chains of fatty acids, the number of double bonds, the viscosity, the acidity and the colour intensity[22].

The absorption at 232 nm permits to evaluate the concentration of Conjugated Diens (CD) produced by the oxidation of the linoleic acid. The CD constitutes, with the hydroperoxides, the primary products of oxidation. As for Conjugated Trienes (CT), they are formed by oxidation of linolenic acid (ω 3). The CT and the secondary oxidation products (aldehydes) resulting from the decomposition of primary products (CD and hydroperoxides) have a maximum absorption near 270 nm[23]. The extinction coefficient values K232 recorded in this study were 1.48,

Table 2. Initial physicochemical characteristics of the study oils and stability to Swift test

Parameters	Oil			
	Argan	**Olive**	**Sun flower**	**Rapeseed**
Peroxide value (meqO2/kg)	0.63 ± 0.11 a	16.33 ± 3.51 d	4.87 ± 0.32 b	7.64 ± 0.24 c
Acidity (%)	0.45 ± 0.04 c	1.02 ± 0.04 d	0.11 ± 0.03 b	0.05 ± 0.01 a
Refraction index	1.470	1.469	1.474	1.473
K 232	1.48	2 .28	3.13	2.86
K 270	0.29	0.14	0.60	0.5
Stability to Swift test (hours)	32.67 ± 1.54 a	26.67 ± 1.15 b	9.67 ± 0.57 d	21.0 ± 1.0 C
Phospholipids (%)	0.86 ± 0.02 a	0.16 ± 0.004 c	0.13 ± 0.006 d	0.23 ± 0.018 B

Values in the same line with different letters are significantly different (P < 0.05)

2.28, 2.86 and 3.13 respectively for the oils of Argan, olive, rapeseed and sunflower. Those of K270 were 0.14, 0.29, 0.51 and 0.60 respectively for the oils of olive, Argan, rapeseed and sunflower (Table 2).

The contents of phospholipids were 0.13, 0.16, 0.23 and 0.86 % for sunflower, olive, rapeseed and Argan oils (Table 2). These results showed that Argan oil was considerably rich in phospholipids in comparison to the other commercial oils. This could be related to the method of extraction used for obtaining Argan oil. Indeed, roasting Argan seeds would allow an increase of the extraction yield of the phospholipids in addition to the formation of the Maillard reaction products in the oil [24,25].

The results of stability, determined by the Swift test, are shown in Table 2. The PV value reached 100 meqO_2/kg after 9.6, 21, 26.67 and 32.67 hours respectively for sunflower, rapeseed, olive and Argan oils. The value of this time is considered a marker of resistance to autoxidation[26]. It appears from these results that the oxidative stability of Argan oil was the most important. This was followed by those of olive, rapeseed and sunflower oils.

The oil storage during ten weeks at 65 °C in presence of air confirmed the oxidation profile obtained by the Swift test. Indeed, the monitoring of four parameters, PV, RI, K232 and K270, showed that oxidation evolved differently according to the type of oil (Figures 1 to 4). The formation of peroxides was fast in sunflower oil, followed by those of rapeseed, olive and Argan oils (Figure 1). Indeed, in sunflower oil, the PV increased in the first week of storage and rapidly reached a maximum value (1000 meqO_2/kg) at week 6. It abruptly decreased to 580 meqO_2/kg at the

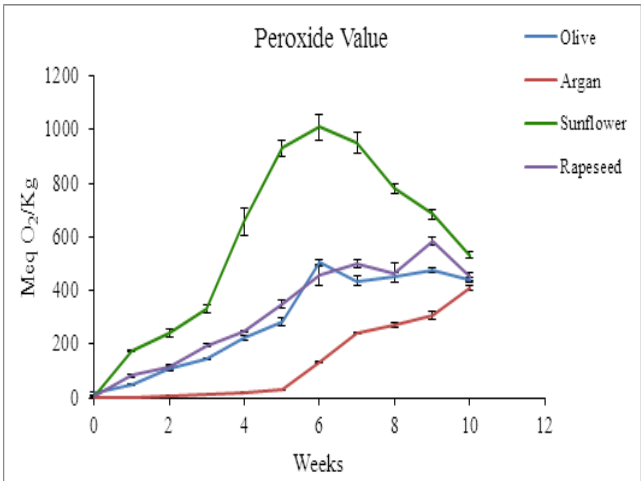

Figure 1. Evolution of peroxide value at 65 °C.

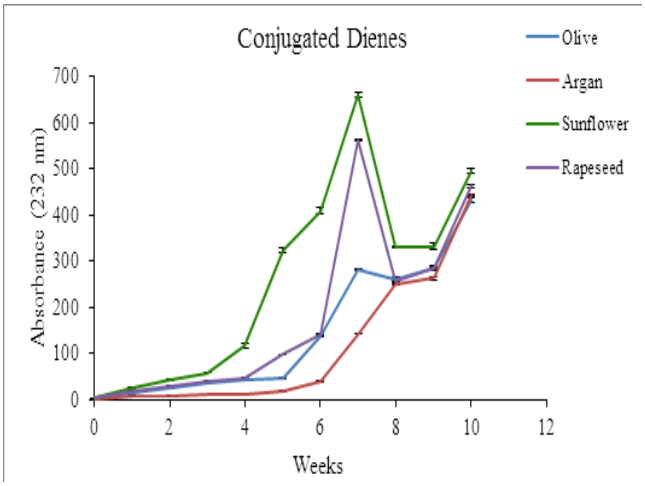

Figure 2. Evolution of conjugated dienes at 65 °C

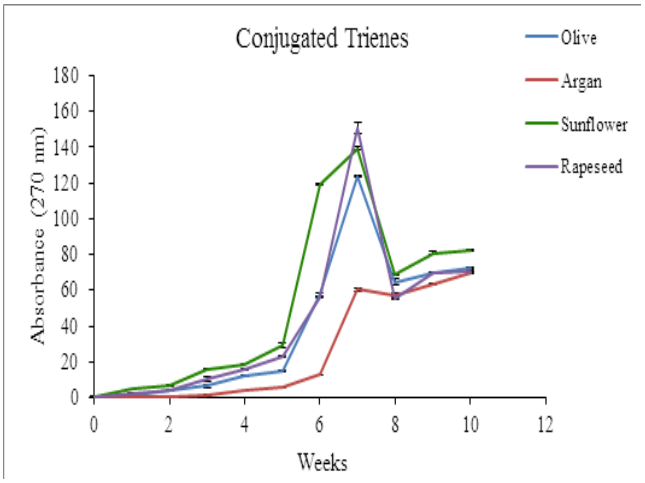

Figure 3. Evolution of conjugated triens at 65 °C

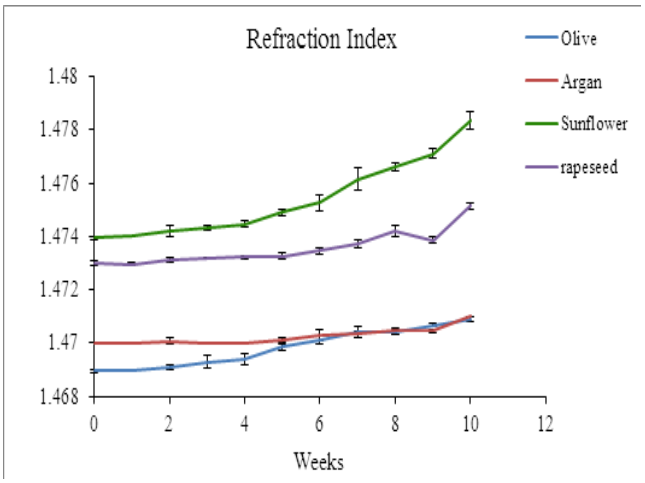

Figure 4. Evolution of refraction index at 65 °C

end of week 10 of storage. In rapeseed and olive oils, PV increased respectively from week 2 and 3 and reached 420 and 480 meqO_2/kg at the end of storage. About Argan oil, the PV remained close to that of the initial state during the first 5 weeks of storage. It increased from week 6 and reached 400 meqO_2/kg at week 10. In general, the time between the start of storage and the sudden increase of the PV correspond to the induction period[25]. Meanwhile, antioxidants contained in the oil protect the fatty acids against the oxidation[17]. Increasing PV at the end of this period reflects the production of hydroperoxides in response to fatty acid oxidation. Hydroperoxides are unstable at high temperatures and decompose resulting in a rapid decreasing of PV. The induction time recorded in this study leads to conclude that Argan oil is more stable (6 weeks) compared to the oils of olive (3 weeks), rapeseed (2 weeks) and sunflower (1week).

As regards the evolution of the absorbance at 232 and 270 nm, the results showed a gradual increase in all commercial oils during storage at 65 °C (Figures 2 and 3). The absorbance at 232 nm was low at the beginning of storage. It increased subsequently to the formation of CD after three, four, five and six weeks respectively in sunflower, rapeseed, olive and Argan oils (Figure 2). The maximum value was reached in the oils of sunflower and rapeseed after seven weeks. However, in olive and Argan oils, the increase in absorbance at 232 nm was characterized by a slow evolution throughout the storage period (Figure 2). The strong decrease in the K232 value is explained by the instability of the CD that turn into secondary oxidation products[27].

Figure 3 showed that K270 increased rapidly after week 5 for sunflower and rapeseed oils, and from week 4 for olive and Argan oils. The elevation of the absorbance at 270 nm is due to the gradual generation of the secondary oxidation products formed by decomposition of the hydroperoxides. The maximum value of K270 recorded for the rapeseed oil was higher than that of sunflower oil. This result is explained by the higher content of conjugated trienes formed during the oxidation of oils rich in linolenic acid such as rapeseed oil[28].

Figure 4 showed that the RI increased during storage. The increase began after week 4 for sunflower and olive oils with faster progression to the first oil. In contrast, Argan and rapeseed oils recorded an elevation of the RI after six weeks of storage. Similar studies previously conducted on soybean, rapeseed and sunflower oils, showed that the increase of the RI depended on the length of exposure to high temperature[29,30]. Other works reported that this index also increased during long-term storage at room temperature[31]. This increase was related to the accumulation of oxidation products such as polymers and polar compounds[29].

Our study showed that the commercial Argan oil had a higher oxidative stability. This was followed by olive, rapeseed and sunflower oils. These results are consistent with other studies which reported that Argan oil was provided with a high thermal stability at 60 °C or even frying, compared with other vegetable oils such as sunflower, rapeseed and olive oils[32,33]. The difference in stability between the studied oils is related to their content in polyunsaturated fatty acids (linoleic and linolenic acids) and antioxidants. Indeed, the polyunsaturated fatty acids with a high number of double bonds, are more susceptible to oxidation reactions that saturated or monounsaturated fatty acids[22,34,35]. In this regard, several studies have demonstrated the value of an enrichment of oils in monounsaturated fatty acids to improve the oxidative stability[30,36]. Similarly, the presence of antioxidants (tocopherols and polyphenols) in vegetable oils allows preventing or stopping the free radical chain reactions that occur during oxidation[28,37-39]. In the case of oils selected for this study, the composition of saturated, monounsaturated and polyunsaturated fatty acids, and the tocopherols content are different. Accordingly, the low stability of sunflower oil obtained in this study could be related to its higher initial content of polyunsaturated fatty acids (61.3%) compared with those of Argan (37%), rapeseed (32%) and olive oils (12.7%). This, despite its higher concentration in tocopherols (500 to 800 mg/kg) compared to the other oils. These results are in agreement with previous works showing that sunflower oil having higher polyunsaturated fatty acid content than that of rapeseed oil, oxidizes more quickly during heating[40,41]. In the case of olive oil, the oxidative stability was related to its richness in antioxidants (tocopherols and phenolic compounds) combined with a low content of polyunsaturated fatty acids[35,39]. In contrast, Argan oil is distinguished from all the tested commercial oils for its high oxidative stability which may be related to a high tocopherols content, and lower level of polyunsaturated fatty acids in comparison to sunflower and rapeseed oils. Furthermore, Argan oil is relatively rich in phospholipids. These possess antioxidant properties and effectively act in

synergy with tocopherols at high temperature[42-43]. Finally, other molecules could contribute to the stability of Argan oil. In fact, the seed roasting during the extraction process would generate diverse products of the Maillard reaction. These compounds are also known for their antioxidant effects[35,44].

In general, the oxidative stability is a qualitative characteristic for predicting the shelf life of food. In fact, during storage, the lipid oxidation in food is slow processes that can take place in several weeks or even months. Therefore, the accelerated oxidation tests are often used in industrial environments such as quality control, in order to predict the progress of oxidation reactions in food and/or compare the susceptibility of lipids to oxidation during storage. In the case of commercial oils, the exposure to a periodic illumination, the nature of storage containers and the presence of oxygen are all factors of deterioration during long-term storage[45]. In this regard, our study showed that the Argan oil could be kept longer in the same storage conditions as other commercial oils.

4. Conclusion

Argan oil showed a higher oxidative stability compared to other commercial oils, thanks both to its fatty acid composition and richness in antioxidants such as tocopherols and phospholipids. The Maillard reaction products formed by roasting the kernels during extraction may contribute to this stability. This would allow a longer shelf life and healthy culinary use, as cooking oil, compared to the other commercial oils.

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