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Influence of roasting temperature and time on the oxidative stability of argan (*Argania spinosa* L.) oil

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Abstract

The present work aimed to evaluate the oxidative stability of argan oil extracted from almonds roasted at different temperatures (75 - 175°C) and durations (10 - 30 min). Oxidative stability was determined by monitoring three oxidation parameters (PV, K_{232}, and K_{270}) during storage at 65°C for a period of ten weeks. The roasting induced slight variations in physicochemical parameters of the oil (peroxide value and absorbance at 232 and 270 nm). The total tocopherols content significantly decreased with increasing roasting temperature (from 970.1 mg/kg in control, to 301.2 mg/kg in oil roasted at 175°C/10 min). On the other hand, the total tocopherols significantly increased with roasting time (505.3 mg/kg in oil roasted at 175°C/30 min). Similar variations were noted for the different forms of tocopherols (α-, γ-, and δ-tocopherol). Phospholipid contents significantly increased as a function of both the temperature and time of roasting (from 0.20 for control, to 1.37% in oil roasted at 175°C/30 min). The lowest oxidation rate was obtained at roasting temperature of 175°C. In addition, the oxidation rate decreased when the roasting time increased from 10 to 30 min. Thus, the best oxidative stability of argan oil was recorded at 175°C/30 min. The improvement in stability under this roasting condition would be due to the increase in the content of oil in phospholipids, and the formation of Maillard reaction products which are endowed with antioxidant activity. In a nutshell, roasting makes it possible to increase the stability of argan oil despite the loss of tocopherols. The condition of 175°C/30 min was demonstrated as the best roasting condition.

Introduction

Oxidation is a major problem in the conservation of edible oils. The roasting of oilseeds improves the oxidative stability of several edible oils. This effect remains largely related to roasting temperatures and durations (Lee *et al.,* 2004; Cai *et al.,* 2013; Siger *et al.,* 2015). In fact, roasting makes it possible to improve the extraction of antioxidant substances such as phenolic compounds, tocopherols, and phospholipids (Wijesundera *et al.,* 2008; Vaidya and Choe, 2011; Jannat *et al.,* 2013). Roasting also leads to the formation of Maillard reaction products endowed with antioxidant activity (Cisneros *et al.,* 2014; Chirinos *et al.,* 2016). On the other hand, tocopherols which are powerful antioxidants are thermosensitive. Their concentration could then decrease or increase according to the temperature and duration of roasting (Yoshida and Takagi, 1997; Lee *et al.,* 2004; Jannat *et al.,* 2013).

Argan oil, extracted from the seeds of *Argania spinosa* L., is widely used in human nutrition, thanks to its high content of polyunsaturated fatty acids and antioxidants. Its extraction, accomplished using mechanical presses or traditionally by manual kneading with water, requires prior roasting of the argan almonds for the development of the organoleptic characteristics appreciated by the consumers. Roasting is traditionally carried out by using wood fire or gas roasters until browning of almonds is achieved. In these conditions, the roasting temperature and time are not controlled, which can lead to oils with varying chemical compositions and oxidative stability.

The studies conducted on the oxidative stability of edible argan oil did not take into account the combined effects of temperature and time of roasting, and the temperature used for roasting was limited to 110°C (Matthäus *et al.,* 2010; Gharby *et al.,* 2011; Harhar *et al.,* 2011; Zaanoun *et al.,* 2014), except for the work by Demnati *et al.* (2018), in which the temperatures were between 150 and 200°C for durations of 10 to 50 min. The present work aimed at determining the roasting conditions for obtaining optimum stability of argan oil without compromising its nutritional value. By doing so, we undertook a study on the oxidative

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stability of argan oils extracted from almonds roasted during 30 min at temperatures of 75, 125, and 175°C. For the roasting temperature of 175°C, two additional durations were considered, namely 10 and 20 min. The oxidative stability was assessed by measuring peroxide value (PV) and absorbance at 232 nm (K*232*) and 270 nm (K*270*) of oils stored using Schaal oven technique (at 65°C) for ten weeks.

Materials and methods

Chemicals and reagents

Tocopherol homologues were purchased from Sigma-Aldrich (St Louis, USA). Chloroform, cyclohexane, hexane, and tetrahydrofuran were purchased from Merck (Darmstadt, Germany). All other chemicals and solvents were of analytical grade, unless otherwise stated.

Oil preparation

The argan fruits were collected in the forest of Admine (Agadir), pulped, and manually crushed. The almonds obtained (300 g) were uniformly spread on a stainless-steel plate. Then, they were roasted using an electric convection oven (Binder ED 115, USA) under continuous aeration for 30 min at 75, 125, and 175°C, and for 10 and 20 min for the temperature of 175°C. After cooling, the almonds were subjected to cold extraction by screw press (Komet S87G press, Germany). The control oil was extracted from unroasted almonds. The oils obtained were then decanted and immediately analysed to determine their initial chemical composition.

Oxidative stability

The oxidative stability of argan oil was determined under accelerated oxidation conditions. The oils samples were distributed in beakers (100 mL) at the rate of 60 g/beaker. The beakers were then kept open in the dark in an oven at 65°C, under continuous aeration for a period of ten weeks. Analyses were carried out every week for the determination of the oxidation parameters, namely the peroxide value (PV) and the UV absorbance at 232 nm (K_{232}) and 270 nm (K_{270}).

Determination of physicochemical parameters

The acid value (AV) and the peroxide value (PV) were determined according to IUPAC 2.201 and 2.501, respectively (Paquot, 1979a; 1979b). The absorbance at 233 and 270 nm was measured using a Varian DMS 80 spectrophotometer after diluting oil samples in cyclohexane (1/100, v/v).

Determination of tocopherols

The tocopherols were analysed using High Performance Liquid Chromatography (Flexar, Perkin-Elmer, USA). The apparatus was equipped with a C_{18} column (20 mm length, 4 mm internal diameter, and 5 µm particle size; Varian Inc.) and a spectrofluorometer detector. Excitation and detection wavelengths were 290 and 330 nm, respectively. The mobile phase was a mixture of tetrahydrofuran:hexane (2:98, v/v), and the flow rate was 1 mL/min.

Determination of phospholipids

The phospholipids were estimated according to AOCS (2009). The oil sample was washed in the presence of zinc oxide. The phosphorus was determined as a blue phosphomolybdic acid complex (Varian DMS 80 spectrophotometer). The total phospholipids were calculated by multiplying the phosphorus content by 25.

Statistical analysis

Data obtained from three replicates ($n = 3$) were processed using the Statistica 6 software. Significant differences were determined using analysis of variance (ANOVA) and the Newman-Keuls method.

Results and discussion

Physicochemical parameters, tocopherol, and phospholipid contents

Table 1 shows the initial physicochemical parameters of the oils extracted from roasted and unroasted almonds. For the control oil, the PV was 0.1 mEq O_2/kg. It slightly increased with the temperature and time of roasting. A maximal value of 0.16 mEq O_2/kg was reached for the roasting condition of 175°C/30 min. Similarly, slight variations in acidity were recorded. The acidity increased by 0.18% in control, to 0.30% for the roasting condition of 175°C/30 min. As for the UV absorbance parameters, the values obtained after roasting showed significant variations with respect to the control ($p < 0.05$). They ranged between 0.81 and 0.87 for K_{232} and between 0.05 and 0.06 for K_{270}. These results agree with previous works on argan oil (Belcadi-Haloui *et al.,* 2018; Demnati *et al.,* 2018) which showed that argan oils extracted from almonds roasted at different temperatures and times were within the range set by the Moroccan standard for extra virgin argan oil (Rahmani, 2005).

Figure 1 also shows the effect of roasting on tocopherol contents. In argan oil extracted from unroasted kernels, the total tocopherols were 970.1 mg/kg, of which γ-tocopherol was 89.4%,

Table 1. Initial physicochemical parameters of argan oils extracted from unroasted and roasted kernels.

Parameter	Temperature/time					
	Control	75°C	125°C	175°C		
		30 min	30 min	10 min	20 min	30 min
PV (mEq O_2/Kg)	0.10 ± 0.01^{a}	0.11 ± 0.01^{b}	0.14 ± 0.01^{d}	0.15 ± 0.02 ^{d}	0.15 ± 0.02^{d}	0.16 ± 0.02^{d}
Acidity (%)	0.18 ± 0.02^{a}	0.24 ± 0.01^{bcde}	0.22 ± 0.01^{b}	0.28 ± 0.01^{fg}	0.28 ± 0.01^{fg}	0.30 ± 0.01^{g}
K_{232}	0.84 ± 0.02^{d}	0.83 ± 0.09^{f}	0.83 ± 0.09^{e}	0.87 ± 0.02^{e}	0.82 ± 0.03^{b}	0.81 ± 0.09^{a}
K_{270}	0.054 ± 0.001^{de}	0.062 ± 0.002^{g}	0.052 ± 0.003^{cde}	0.055 ± 0.002^{c}	0.053 ± 0.003^{cde}	0.052 ± 0.004^{cde}

Values are means ± standard deviations from triplicate measurements (n = 3). Means within the same row followed by different letters are significantly different ($p < 0.05$).

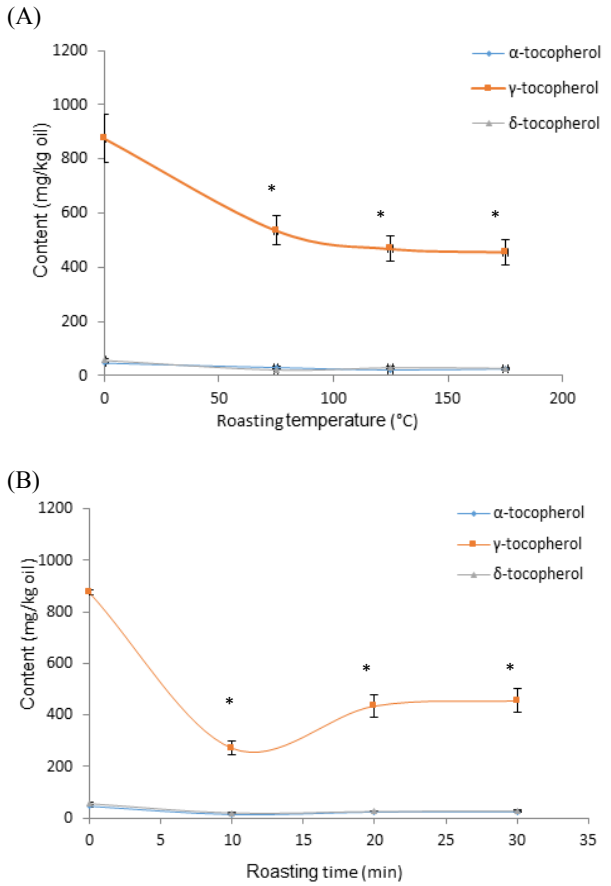


Figure 1. Variation of tocopherol contents based on the roasting temperature (A) and time (B). Values are means ± standard deviations from triplicate measurements ($n = 3$). * = Significantly different from control ($p < 0.05$).

α- tocopherol was 4.8%, and δ-tocopherol was 5.8%. Roasting caused a significant decrease ($p < 0.05$) in tocopherol levels as a function of temperature. The decrease of total tocopherol contents was 40, 48, and 49% after roasting at 75, 125, and 175°C/30 min, respectively. The different forms of tocopherols showed similar reductions. On the other hand, the content of total tocopherols significantly increased with the duration of roasting at 175°C. It varied from 305.1 to 505.3 mg/kg when the roasting time increased from 10 to 30 min. The content of α-, γ-, and δ-tocopherols increased respectively from 14.3 to 25.2 mg/kg, 387.3 to 455.5 mg/kg, and 19.8 to 27.1 mg/kg. Several studies have reported changes in tocopherol contents due to the roasting of different oilseeds. The decrease in the tocopherol contents was explained by their thermo-sensitivity, thus resulting in their destruction by heating. Their increase was attributed to better extractability following the destruction of oilseed tissue structures and the breakdown of their binding to membrane proteins and phospholipids (Moreau *et al.,* 1999; Vaidya and Choe, 2011).

Figure 2 shows the effects of roasting on phospholipid contents. The conditions of 75°C/30 min and 125°C/30 min did not induce significant changes. On the other hand, 175°C led to an increase in the phospholipid contents. In the same way, the phospholipid contents significantly increased with the roasting time at 175°C. It reached 1.37% after roasting for 30 min at 175°C (0.20% for the control). Several studies reported an increase in the phospholipid contents of argan oil obtained from roasted oilseeds (Matthäus *et al.,* 2010; Harhar *et al.,* 2011; Belcadi-Haloui *et al.,* 2018). The authors opined this to be due to better extraction rate of phospholipids following the destruction of the argan kernel tissues by heating.

Oxidative stability

Figure 3 shows the evolution of the PV in the oil samples stored at 65°C. The kinetics were characterised by three distinct phases; first where the PV increase was low, followed by a second period where the PV rapidly increased, and a last step where the value decreased. The period between the initial state and the beginning of the rapid increase of the PV corresponded to the induction time of the oxidation, which was proportional to the oil stability. In the present work, the induction period was two weeks for the control, three weeks for the oils from almonds roasted at low temperatures (75 and 125°C), and five weeks for the oil from almonds roasted at 175°C. The

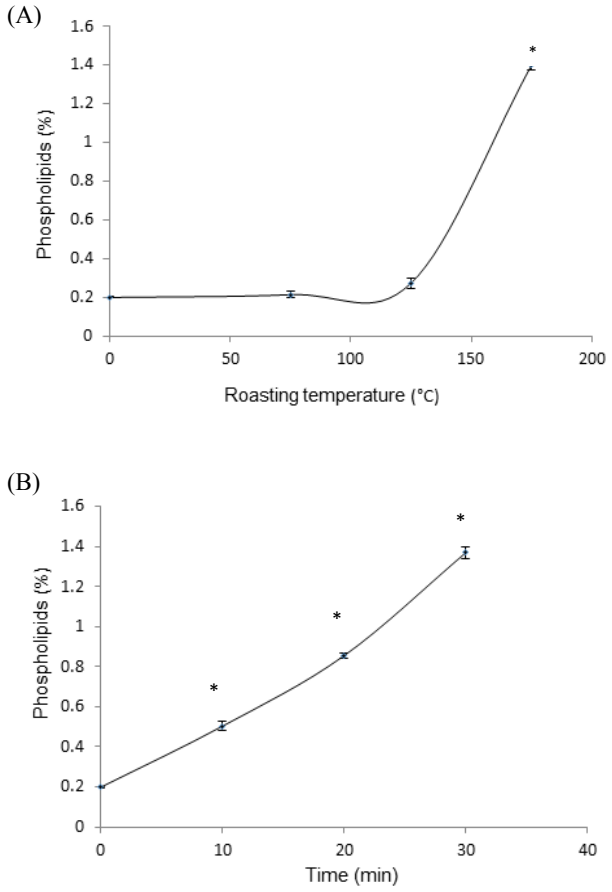
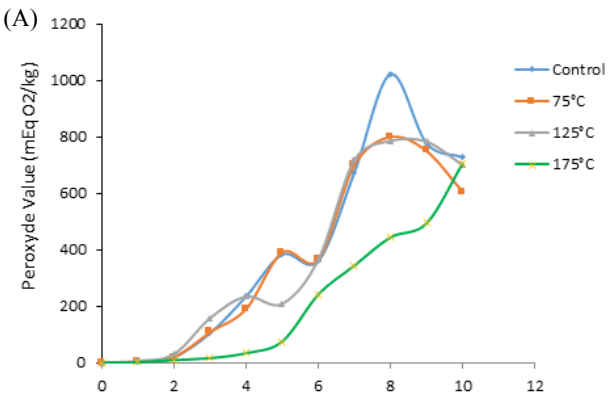


Figure 2. Variation of phospholipid contents based on the roasting temperature (A) and time (B). Values are means ± standard deviations from triplicate measurements (n = 3). * = Significantly different from control (p < 0.05).

oxidative stability also increased with the duration of roasting at 175°C. Thus, the induction time of two weeks for the control increased to three weeks for the roasting times of 10 and 20 min, and reached five weeks for the duration of 30 min. The highest values of the PV were reached after eight weeks for the control and the oils obtained after roasting at 75 and 125°C. This period exceeded ten weeks when the roasting temperature was 175°C. At week 8, the PV reached 1022.22 mEq O_2/kg for the control, and only 444.44 mEq O_2/kg for the oil obtained after roasting at 175°C.



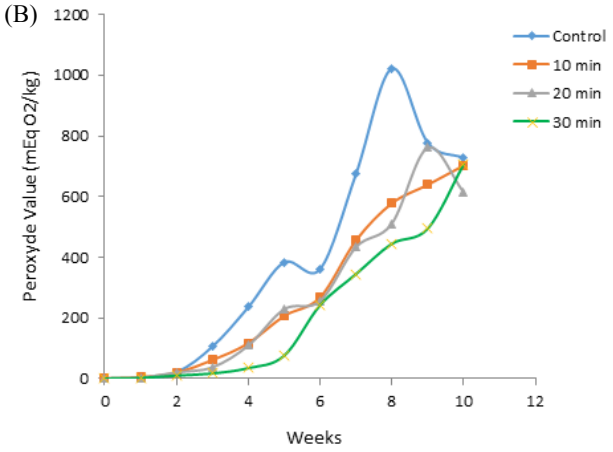


Figure 3. Effect of storage at 65°C on peroxide value of argan oils extracted from kernels roasted at different temperatures (A) and times (B).

Figure 4 reports the evolution of K_{232}. This parameter makes it possible to evaluate the content of the oil in conjugated dienes (CD). The recorded values remained low during the six weeks of storage. They gradually increased until the end of storage. The formation of CD increased during storage in parallel with the increase of hydroperoxides

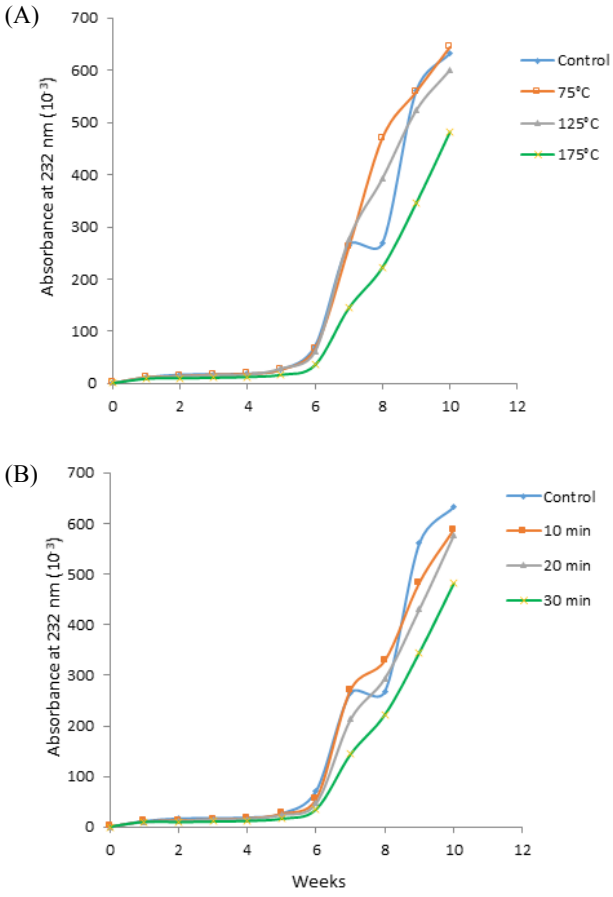


Figure 4. Effect of storage at 65°C on K_{232} of argan oils extracted from kernels roasted at different temperatures (A) and times (B).

evaluated by PV. Their formation rate was slower at 175°C as compared to 75 and 125°C, and the control. CD and hydroperoxides represent the primary products of the oxidation of unsaturated fatty acids. K_{270} makes it possible to assess the content in secondary oxidation products (aldehydes and ketones) resulting from the decomposition of hydroperoxides. It is a parameter for evaluating the degree of sensory alteration of the oil. The results showed that K_{270} rapidly increased between week 6 and 7, then decreased from week 7 to week 8, and increased again from week 8 (Figure 5). These fluctuations could be explained by the presence of the antioxidants in the oil, in particular the tocopherols. In fact, the antioxidants stabilise the hydroperoxides and inhibit their degradation in secondary products. The evolution of K_{270} was less rapid in oils extracted from almonds roasted at high temperatures (175°C) as compared to control and oils from roasting at low temperatures (75 and 125°C). K_{270} also depended on roasting duration. The values obtained at weeks 9 and 10 were lower for the duration 30 min as compared to 10 and 20 min, and the control.

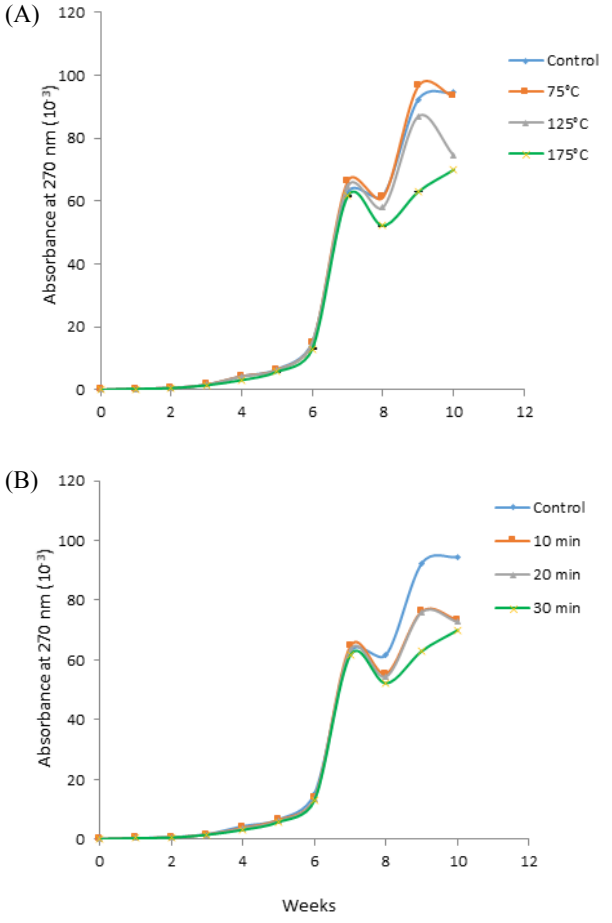


Figure 5. Effect of storage at 65°C on K_{270} of argan oils extracted from kernels roasted at different temperatures (A) and times (B).

The favourable effect of roasting on oxidative stability has been widely documented for other edible oils. Several authors reported that both roasting temperature and duration increased the stability of the oil (Lee *et al.,* 2004; 2015; Spielmeyer *et al.,* 2009; Vujasinovic *et al.,* 2012; Siger *et al.,* 2015). These effects were explained by the increase in the content of oils in natural antioxidants such as tocopherols, phospholipids, polyphenols, and phytosterols. Indeed, the extraction of antioxidants would be improved by the destruction of tissue structures in roasted kernels. In addition, roasting is responsible for the formation of Maillard reaction products that enhance the effect of antioxidants naturally present in edible oils (Cisneros *et al.,* 2014; Siger and Michalak, 2016). The products of the Maillard reaction result from the condensation of reducing sugars with nitrogenous substances (proteins and free amino acids). Zhang *et al.* (2019) reported a decrease in the content of pepper seeds in simple reducing carbohydrates (e.g. glucose and mannose) and free amino acids with the increase of roasting temperature. This shows that high temperatures produce more Maillard reaction products, which results in better oil stability. In fact, it has been shown that the products of the Maillard reaction inhibit the formation of hydroperoxides and their decomposition into carbonyl compounds (Kirigaya *et al.,* 1968). In addition, some of these products such as melanoidins prevent the degradation of tocopherols by retaining them in their structure (Vaidya and Choe, 2011; Vaidya and Eun, 2013). Concerning argan oil, studies devoted to the effect of roasting on the oxidative stability are not numerous. In addition, the roasting temperature was limited to 110°C, except for the work of Demnati *et al.* (2018) which used roasting conditions comparable to ours (temperatures of 150, 175, and 200°C for periods of 10, 30, and 50 min). Our results are in agreement with those reported by these authors who concluded that the increase in temperature and duration of roasting caused a rise in the phospholipid levels and an improvement in the oxidative stability of the argan oil. Our results also showed that the use of low temperatures could be compensated by an increase in roasting time.

In the present work, the best oxidative stability of argan oil was achieved at 175°C/30 min despite the lower tocopherol contents as compared to the unroasted sample. This suggests that improving the stability of the oil does not solely depend on the content of tocopherols. Phospholipids whose content increased with temperature and roasting time, seemed to play an important role. In fact, phospholipids are able to chelate metals and act in synergy with

tocopherols (Judde *et al.,* 2003; Vujasinovic *et al.,* 2012; Shrestha and De Meulenaer, 2014). They also prevent the decomposition of hydroperoxides occurring at roasting temperatures (Alam *et al.,* 1997). Finally, other compounds present in argan oil such as polyphenols and Maillard reaction products have antioxidant activity. Nevertheless, further studies are warranted which would elucidate the involvement of these antioxidants in the protection of argan oil against the oxidation.

Conclusion

The present work showed that the roasting of argan almonds led to a reduction in the content of tocopherols and an increase in that of phospholipids. Roasting also resulted in an increase in the oxidative stability of the oil. This effect could be explained by the synergistic antioxidant action of phospholipids, tocopherols, and products of the Maillard reaction in addition to other antioxidants such as sterols and polyphenols which are naturally present in the oil. The present work also demonstrated that the oxidative stability of the oil depends on the interaction of both temperature and time. In this respect, the combination temperature/time of 175°C/30 min yielded the best oxidative stability without compromising the quality of the argan oil as extra virgin oil.

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