

## Research Article

# Oxidative Stability at Different Storage Conditions and Adulteration Detection of Prickly Pear Seeds Oil

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Lipid oxidation and adulteration have a negative impact on functionality and notoriety of foods especially vegetable oils and cause economic losses. The present study investigates the control of two commercial quality aspects of prickly pear seeds oil (PPSO): oxidative stability during storage and detection of adulteration. Peroxide index, specific extinction coefficients  $K_{232}$  and  $K_{270}$ , free acidity, and fatty acids composition were evaluated during different periods of incubation (6, 12, and 18 months) at various temperatures (4°C, 25°C, 40°C, and uncontrolled room temperature ranging between 4°C and 40°C) with different packaging (protected and unprotected from sunlight, with and without nitrogen gas bubbling). Based on the physicochemical and biochemical parameters evolution, this study has shown that PPSO stored at 4°C for 18 months preserves the initial quality. However, at 40°C, an intense lipid oxidative process occurred after 6 months of storage. The changes have also affected fatty acids composition, especially rates of linoleic and oleic acids. The shelf-life of oils stored at 25°C and at uncontrolled room temperature can be limited to 6 months. Regarding the impact of light and nitrogen bubbling, sunlight has affected seriously the oxidative stability of oils after 12 months of storage and the bubbling with nitrogen has improved their stability when they have been stored in clear glass bottles. The levels of adulteration detection using fatty acids as markers are relatively high. The detection of oil adulteration can be depicted by fatty acids composition up to 15% of olive and almond oils and up to 20% of rapeseed oil. The iodine value could also be an indicator of the sunflower oil presence in PPSO. Therefore, other minor compounds including sterols and tocopherols should be investigated to depict PPSO adulteration with cheaper oils and to determine lower levels of detection in order to ensure the authenticity of PPSO.

## 1. Introduction

In Morocco, as well as in the Mediterranean countries, prickly pear (*Opuntia* spp.) has been considered as an interesting raw material for food industries. Efforts have been made to develop new industrial products from agro-wastes, among them, the production and marketing of seeds oil. The chemical composition of prickly pear seeds oil (PPSO) was widely studied [1, 2]. Seeds constitute more than 15% of the fruit mass and their oil represents 10–15% of total seeds mass [3]. This oil has a high content of unsaturated fatty acids

ranging from 73.5% to 88.3% [3, 4]. Linoleic acid is the major fatty acid followed by oleic acid. This oil has also high saponification value (186.63 mg KOH/g of oil) and important contents of tocopherols (up to 94.60 mg/100 g) and sterols (90 mg/kg) [4, 5].

The major tocopherol of PPSO is gamma-tocopherol, representing an average of 90% of total tocopherols, compared with delta-tocopherol (9%) and alpha-tocopherol (1.8%) [6]. These tocopherol compounds are used in food, cosmetics, and pharmaceutical industries [7]. Alpha-tocopherol is recommended for human and

animal consumption because it has a higher biological activity than other tocopherols, but gamma-tocopherol shows a higher antioxidant capacity as compared to alpha-tocopherol [8]. According to the previous studies [3–5], the PPSO can reduce cholesterol, especially low and very low density lipoproteins (LDL and VLDL) levels. PPSO was also studied as dietary supplement for animals at the rate of 25 g/kg which has reduced feed conversion efficiency [9]. As an added function, PPSO has also shown anticorrosion capacity inhibiting corrosion of mild steel in 1M HCl [10].

However, there are not many studies concerning the oxidative stability of oils in general and even less for PPOS in particular. It has been reported among the few ones that the stability to oxidation of PPSO is lower than those of olive oil and argan oil, due to the PPSO high level of unsaturated fatty acids [4]. Nevertheless, as far as we know, the oxidative stability of PPSO during storage at different conditions has not been reported. Overall, lipid oxidation has a negative impact on functional, sensory, and nutritional qualities of food and causes economic losses [11, 12]. The oxidative processes are the same for all fats and oils, while the reactions rates are different [13].

In addition to the oxidative stability challenge, the detection of oil adulteration especially in cosmetics field is also an important quality aspect to ensure an authentic product especially for oils with high marketing value as is the case with PPSO. The adulteration of PPSO with relatively low-price vegetable oils is common, and, consequently, there is an urgent need to control quality aspects of this oil. Furthermore, there is a need for reliable, rapid, and inexpensive adulteration detection methods in the commercial oil industry [14]. Several methods have been proposed for the detection of the adulteration of high quality oils such as high performance liquid chromatography (HPLC) [15], solid-phase microextraction-gas chromatography with flame ionisation detection (SPME-GC-FID), electronic nose, and direct SPME mass spectrometry (MS) combined with principal component analysis (PCA) [16].

To the best of our knowledge, this study is the first investigation associating two aspects relating to the PPSO preservation and adulteration which have an immense importance for sanitary and commercial quality, making it possible for the consumer as well as the manufacturer and the authority, to educate the optimal conditions of typicity and authenticity preservation, to raise awareness about potential adulteration, and to pave the way for the establishment of a commercial quality standard of this oil.

In this context, this work aims (i) to study the oxidative stability of PPSO by evaluating its chemical composition changes at different temperatures (4°C, 25°C, 40°C, and uncontrolled room temperature) and conditions of storage (protected and unprotected from sunlight and oxygen) during 18 months and (ii) to detect levels of oil adulteration with comparatively four cheaper vegetables oils (sunflower, rapeseed, olive, and almond oils).

## 2. Materials and Methods

**2.1. Plant Material and Seeds Oil Extraction.** The study was conducted on the variety of prickly pear (*Opuntia ficus-indica* L.) locally called “Mles” or “Safra” in Rhamna area (center of Morocco). A plant voucher specimen was deposited at the Regional Herbarium “MARK” at Cadi Ayyad University in Marrakesh. The fruits were harvested at full maturity stage. Immediately after harvest, seeds were separated from the juicy pulp, washed, and dried at room temperature. The extraction of the oil was carried out using a mechanical press with endless screw belonging to the Sebbar Rhamna Cooperative.

**2.2. Study Design for the Oxidative Stability Evaluation.** A completely randomized design was used for evaluating the oxidative stability by comparing the quality parameters and fatty acids composition of oil samples (64 oil vials with a capacity of 30 mL) stored at the following conditions: in dark glass bottles at 4°C, 25°C, and 40°C; in dark glass bottles at uncontrolled room temperature ranging between 4°C and 40°C, without nitrogen gas bubbling; in clear glass bottles at uncontrolled room temperature ranging between 4°C and 40°C, without nitrogen gas bubbling; in dark glass bottles, bubbled with nitrogen gas before sealing and placed at uncontrolled room temperature ranging between 4°C and 40°C; and in clear glass bottles, bubbled with nitrogen gas before sealing and placed at uncontrolled room temperature ranging between 4°C and 40°C. Bubbling with nitrogen gas has been made in order to study the impact of the oxygen on oil oxidation. Clear or dark bottles were used with 2 mL headspace volume. Sampling times were at the beginning of the trial ( $T_0$ ) (as a control) and after 6, 12, and 18 months. Hermetically sealed bottles were used for each test.

**2.3. Quality Parameters.** The determined PPSO quality parameters are peroxide index (meq of  $O_2$ /kg of oil), specific extinction coefficients  $K_{232}$  and  $K_{270}$ , and free acidity (% of oleic acid). The peroxide index was determined according to standard NF 60-220 [17]. The sample is treated in solution with a mixture of acetic acid and chloroform and then with a solution of potassium iodide. The liberated iodine is titrated with a standard solution of sodium thiosulfate using starch as an indicator. According to IOC standard [18], the specific extinction coefficients  $K_{232}$  (or conjugated dienes) and  $K_{270}$  (or conjugated trienes) are absorbances at 232 and 270 wavelengths, respectively, and expressed as specific extinction  $E_{1\%}^{1\text{cm}}$  (extinction of a 1% fat solution in the prescribed solvent, for a thickness of 1 cm) conventionally denoted by K. For free acidity determination, the method used is that described by standard NF.T 60-204 [17]. This method consists in taking 1 g of oil in 20 mL of an equal volume of ether/ethanol (50/50 (V/V)) neutralized and then titration of the free fatty acids using a solution ethanolic potassium hydroxide in the presence of phenolphthalein. The end of the dosage is marked by the appearance of a slightly pink color. The oil color was determined by measuring  $L^*a^*b^*$  parameters of Hunter Lab scale with a

spectrocolorimeter for liquid (Tintometer Lovibond PFX 195). Contents of oil pigments (expressed in ppm) were determined by the methods described by Wolff [19] for chlorophyll, by Psoiadou and Tsimidou [20] for  $\alpha$ -pheophytin, and by Mosquera-Minguez et al. [21] for carotenoids. The fractions of  $\alpha$ -pheophytin and chlorophyll were quantified at the wavelengths 630 nm, 670 nm, and 710 nm and those of the carotenoids at 470 nm.

**2.4. Fatty Acids Composition.** Fatty acids composition was determined by gas chromatography analysis according to the analytical methods described in the IOC standard [22]. The preparation of fatty acid methyl esters (FAME) was carried out by adding 0.1 mL of the methanolic solution of potassium hydroxide (2 N) to the oil solution (0.1 g) purified in 1 mL of n-heptane. Before injection into the chromatograph, this n-heptane solution was shaken vigorously for 15 s and let stand until the upper part becomes clear (5 min). The fatty acids separation was carried out using gas chromatograph Varian CP 3380, equipped with capillary column (CP-Wax 50 CB: L = 25;  $\Phi$  = 0.25 mm; Ft = 0.20  $\mu$ m), using injector split-splitless (split ratio of 1:100) equipped with auto-sampler Varian CP-8400 and FID detector. The temperatures of injector, detector, and oven were 220°C, 230°C, and 190°C, respectively. Nitrogen was used as carrier gas with flow rate of 154.0 mL/min. The injection volume was 1  $\mu$ L. The fatty acids identification was achieved by the use of control fatty acids and also by the recourse to the methods of the imprint. For fatty acids quantification, the total area (TA) is the sum of all the peaks that appear in the chromatogram, from C16:0 to C20:1. The percentage of each peak (FAx (%)) was calculated by using the following equation:

$$FAx (\%) = 100 \left( \frac{Ax}{AT} \right), \quad (1)$$

where Ax is the individual peak area of each FAME and AT is the total area of all FAME peaks.

The iodine value (IV), which measures the level of unsaturation in oils and expressed in gram iodine absorbed by 100 g of oil, was calculated from the percentages of fatty acids according to (2) proposed by Diraman and Dibeklioğlu [23]:

$$IV = (\% \text{ palmitoleic acid} \times 1.001) + (\% \text{ oleic acid} \times 0.899) \\ + (\% \text{ linoleic acid} \times 1.814) + (\% \text{ linolenic acid} \times 2.737). \quad (2)$$

**2.5. Adulteration Test.** In order to detect adulteration of PPSO, four fresh vegetable oils (olive, almond, rapeseed, and sunflower oils) were added to PPSO. These oils, purchased from the market, are characterized by their low commercial values compared to PPSO and the same appearance as PPSO especially olive oil. These oils were mixed with PPSO at different rates ranging between 1% and 50% (vegetable oil/PPSO). The determination of fatty acids profiles of pure oils (as controls) and adulterated oils was

carried out by gas chromatography analysis according to the analytical method previously described.

**2.6. Statistical Analysis.** Data are presented in tables and figures as (means  $\pm$  standard deviations) of three replicates. Analyses of variance especially two-way ANOVA were applied for the oxidative stability tests to assess interaction and main effects of time and temperature and time and packaging conditions for PPSO quality criteria. For the adulteration test, the one-way ANOVA was applied. Student–Newman–Keuls test at level of  $P < 0.05$  was also performed to compare between means and to determine the homogenous groups of means.

### 3. Results and Discussion

**3.1. Effect of Temperature on PPSO Quality during Storage.** The results of the two-way ANOVA, with the objective being the study of the time and temperature interaction effects on the PPSO quality during storage, have shown that all the studied quality parameters (peroxide index, free acidity, and specific extinction coefficients  $K_{232}$  and  $K_{270}$ ) have experienced very high significance difference ( $P < 0.001$ ) (Figure 1). However, for fatty acids composition, very high significance difference ( $P < 0.001$ ) was recorded for the “time \* temperature” interaction only for the three fatty acids C18:0 (oleic acid), C18:1 (linoleic acid), and C18:2 (linolenic acid) from omega 9, omega 6, and omega 3 families, respectively (Table 1). The effect of each studied temperature on the PPSO quality during storage is presented below.

**3.1.1. Effect of Storage at 4°C.** During 18 months of storage at 4°C, PPSO remained stable without significant changes of free acidity, specific extinction coefficients  $K_{232}$  and  $K_{270}$ , and fatty acids composition. Although it is common that, during such long periods of storage, hydroperoxides split into short chain aromatic organic compounds (mainly aldehydes, ketones, alcohols, and short chain fatty acids), which cause the rancidity [24]. At 4°C, the secondary reactions of oxidation were stopped. Only reactions of primary oxidation occurred and they started at 18 months (peroxide index increased from 3.4 to 10.8 meq  $O_2$ /kg of oil after 18 months) (Figures 1(a), 1(c), and 1(d)). As most of the supermarkets and shops operate around 25°C, the low temperatures (close to 4°C) could be used as storage temperatures by consumers after purchasing this oil to extend its shelf-life.

**3.1.2. Effect of Storage at 25°C.** The oxidation of PPSO stored at 25°C was noticeable at 12 months of storage. The production of hydroperoxides and conjugated dienes increased, checked by peroxide index and  $K_{232}$  value (Figures 1(a) and 1(c)). The values of these parameters vary from 3.4 to 30.5 meq  $O_2$ /kg of oil and from 1.65 to 1.96 respectively. After 18 months of storage, the quality of PPSO was strongly affected (Figure 1). There was an increase of the free acidity (from 0.25 to 0.5%), the peroxide index (from 3.4 to 38.83 meq  $O_2$ /kg of oil), and the conjugated trienes (from

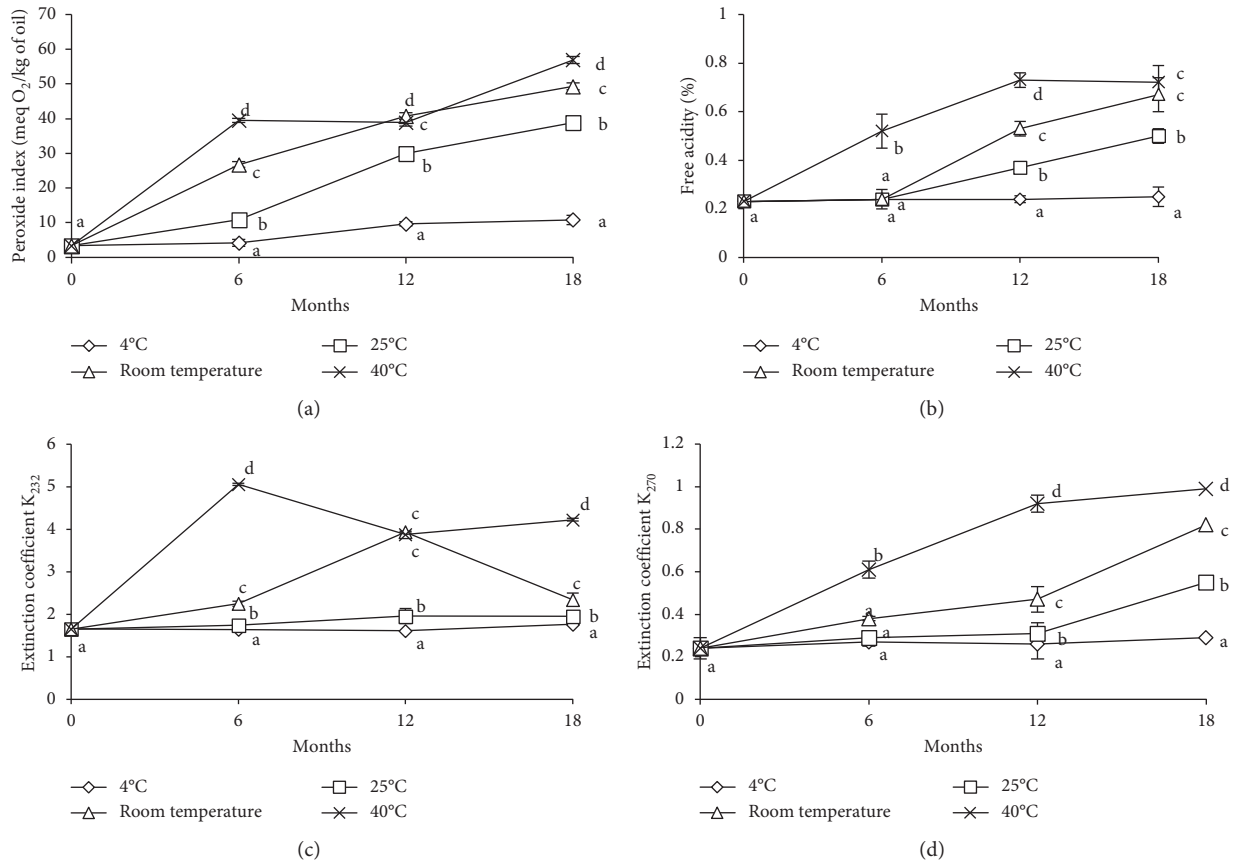


FIGURE 1: Evolution of quality parameters of prickly pear seeds oil during storage at various temperatures: 4°C, 25°C, 40°C, and uncontrolled room temperature. (a) Peroxide index. (b) Free acidity. (c) Specific extinction coefficient  $K_{232}$ . (d) Specific extinction coefficient  $K_{270}$ . The error bars represent the standard deviation of three replicates. Means with the same letter for the same storage time are not significantly different according to Student–Newman–Keuls test at  $P < 0.05$ .

0.24 to 0.55), while  $K_{232}$  remained quite stable and fatty acids composition did not show significant changes (Table 1). The difference between the evolution of peroxide index and  $K_{232}$  value is probably related to the fact that the former measures hydroperoxides, while the latter measures hydroperoxides and conjugated dienes [25].

**3.1.3. Effect of Storage at 40°C.** The storage of PPSO at 40°C impacted seriously the quality of oil. All studied parameters increased from the start of storage, revealing the occurrence of intense oxidative processes. The production of hydroperoxides and conjugated dienes ( $K_{232}$ ) swiftly increased. The peroxide index and conjugated dienes ( $K_{232}$ ) varied from 3.40 to 39.48 meq O<sub>2</sub>/kg of oil and from 1.65 to 5.06, respectively (Figures 1(a) and 1(c)). The two parameters (peroxide index and  $K_{232}$ ) have decreased at 12 months of storage indicating that the rate of hydroperoxides decomposition was high. However, the conjugated trienes production ( $K_{270}$ ) has increased significantly during 12 months and, after this period, it remained stable. The increase of  $K_{270}$  indicates the production of volatile compounds originating from hydroperoxide decomposition during the last phase of oxidation; these compounds are responsible for the rancid defect in oil [26]. The free acidity has increased continuously

to reach at 12 months of storage the maximum value of 0.72% (Figure 1(b)) indicating the accelerated triacylglycerol degradation which contributes in increasing acid levels [27]. In the same way as conjugated trienes ( $K_{270}$ ), free acidity remained stable between 12 and 18 months of storage (Figures 1(b), 1(d)). The occurred changes in PPSO have also concerned fatty acid compositions. From 6 months, significant variations were observed. The changes were detected in the concentrations of monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) especially those of linoleic acid (C18:2) and oleic acid (C18:1). Linoleic acid concentration has decreased from 61.44% to 60.10% (decrease rate of 2.2%) after 18 months. However, the concentration of oleic acid has increased from 21.25% to 22.47% (increase rate of 5.8%) (Table 1). These observations are in agreement with a reported nuclear magnetic resonance study, which has confirmed the fact that fatty acids degradation rate increases with the number of double bonds in the molecule [28].

**3.1.4. Effect of Storage at Uncontrolled Room Temperature.** Uncontrolled room temperature represents the common temperature of storage in local markets. During the study, the room temperature ranged from 4°C (indoor coldest

TABLE 1: Fatty acids composition of prickly pear seeds oil as a function of storage time at various temperatures: 4°C, 25°C, 40°C, and uncontrolled room temperature\*.

Temperature (°C)	Time*** (months)	Fatty acids (%)								
		Myristic acid C14:0	Palmitic acid C16:0	Palmitoleic acid C16:1	Stearic acid C18:0	Oleic acid I	Linoleic acid C18:2	Linolenic acid C18:3	Arachic acid C20:0	Eicosenoic acid C20:1
—	0	0.07 <sup>a</sup> ± 0.04	12.34 <sup>a</sup> ± 0.01	0.74 <sup>a</sup> ± 0.03	3.24 <sup>a</sup> ± 0.05	21.25 <sup>a</sup> ± 0.10	61.44 <sup>a</sup> ± 0.06	0.24 <sup>a</sup> ± 0.07	0.31 <sup>a</sup> ± 0.01	0.29 <sup>a</sup> ± 0.07
4	6	0.06 <sup>a</sup> ± 0.02	12.28 <sup>a</sup> ± 0.01	0.77 <sup>a</sup> ± 0.02	3.25 <sup>a</sup> ± 0.02	21.26 <sup>a</sup> ± 0.03	61.43 <sup>b</sup> ± 0.09	0.22 <sup>a</sup> ± 0.04	0.29 <sup>a</sup> ± 0.01	0.28 <sup>a</sup> ± 0.06
	12	0.07 <sup>a</sup> ± 0.03	12.29 <sup>a</sup> ± 0.06	0.73 <sup>a</sup> ± 0.02	3.26 <sup>a</sup> ± 0.02	21.27 <sup>a</sup> ± 0.03	61.44 <sup>b</sup> ± 0.09	0.26 <sup>a</sup> ± 0.02	0.34 <sup>a</sup> ± 0.06	0.25 <sup>a</sup> ± 0.09
	18	0.05 <sup>a</sup> ± 0.03	12.31 <sup>a</sup> ± 0.11	0.72 <sup>a</sup> ± 0.01	3.26 <sup>a</sup> ± 0.01	21.35 <sup>a</sup> ± 0.18	61.51 <sup>b</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.32 <sup>a</sup> ± 0.06	0.29 <sup>a</sup> ± 0.00
25	6	0.07 <sup>a</sup> ± 0.01	12.26 <sup>a</sup> ± 0.01	0.72 <sup>a</sup> ± 0.01	3.32 <sup>b</sup> ± 0.15	21.24 <sup>a</sup> ± 0.07	61.49 <sup>b</sup> ± 0.09	0.24 <sup>a</sup> ± 0.01	0.27 <sup>a</sup> ± 0.03	0.36 <sup>a</sup> ± 0.05
	12	0.08 <sup>a</sup> ± 0.03	12.33 <sup>a</sup> ± 0.10	0.75 <sup>a</sup> ± 0.01	3.28 <sup>a</sup> ± 0.07	21.31 <sup>a</sup> ± 0.07	61.47 <sup>b</sup> ± 0.10	0.27 <sup>a</sup> ± 0.01	0.28 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.07
	18	0.06 <sup>a</sup> ± 0.01	12.31 <sup>a</sup> ± 0.06	0.73 <sup>a</sup> ± 0.02	3.28 <sup>a</sup> ± 0.07	21.31 <sup>a</sup> ± 0.07	61.49 <sup>b</sup> ± 0.08	0.25 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.01	0.27 <sup>a</sup> ± 0.04
40	6	0.08 <sup>a</sup> ± 0.01	12.31 <sup>a</sup> ± 0.04	0.76 <sup>a</sup> ± 0.00	3.28 <sup>ab</sup> ± 0.04	21.48 <sup>b</sup> ± 0.03	61.02 <sup>a</sup> ± 0.07	0.26 <sup>a</sup> ± 0.00	0.26 <sup>a</sup> ± 0.00	0.34 <sup>a</sup> ± 0.01
	12	0.07 <sup>a</sup> ± 0.01	12.40 <sup>b</sup> ± 0.05	0.74 <sup>a</sup> ± 0.02	3.31 <sup>a</sup> ± 0.01	21.49 <sup>b</sup> ± 0.07	61.05 <sup>a</sup> ± 0.09	0.24 <sup>a</sup> ± 0.01	0.26 <sup>a</sup> ± 0.03	0.29 <sup>a</sup> ± 0.05
	18	0.09 <sup>a</sup> ± 0.03	12.40 <sup>a</sup> ± 0.05	0.71 <sup>a</sup> ± 0.02	3.62 <sup>b</sup> ± 0.01	22.47 <sup>b</sup> ± 0.07	60.10 <sup>a</sup> ± 0.13	0.24 <sup>a</sup> ± 0.01	0.31 <sup>a</sup> ± 0.00	0.28 <sup>a</sup> ± 0.06
RT**	6	0.05 <sup>a</sup> ± 0.02	12.34 <sup>a</sup> ± 0.01	0.75 <sup>a</sup> ± 0.03	3.24 <sup>a</sup> ± 0.05	21.25 <sup>a</sup> ± 0.10	61.42 <sup>b</sup> ± 0.05	0.26 <sup>a</sup> ± 0.01	0.29 <sup>a</sup> ± 0.00	0.33 <sup>a</sup> ± 0.05
	12	0.05 <sup>a</sup> ± 0.02	12.29 <sup>a</sup> ± 0.08	0.74 <sup>a</sup> ± 0.00	3.29 <sup>a</sup> ± 0.06	21.27 <sup>a</sup> ± 0.07	61.56 <sup>b</sup> ± 0.12	0.26 <sup>a</sup> ± 0.03	0.29 <sup>a</sup> ± 0.01	0.27 <sup>a</sup> ± 0.02
	18	0.07 <sup>a</sup> ± 0.03	12.28 <sup>a</sup> ± 0.04	0.75 <sup>a</sup> ± 0.00	3.34 <sup>a</sup> ± 0.08	21.28 <sup>a</sup> ± 0.03	61.42 <sup>b</sup> ± 0.12	0.23 <sup>a</sup> ± 0.04	0.30 <sup>a</sup> ± 0.01	0.28 <sup>a</sup> ± 0.05

\* Means with the same letter within the column for the same storage time are not significantly different according to Student–Newman–Keuls test at  $P < 0.05$ . \*\* RT: uncontrolled room temperature ranging between 4°C and 40°C. \*\*\* Time 0 corresponds to the beginning of the trial ( $T_0$ ).

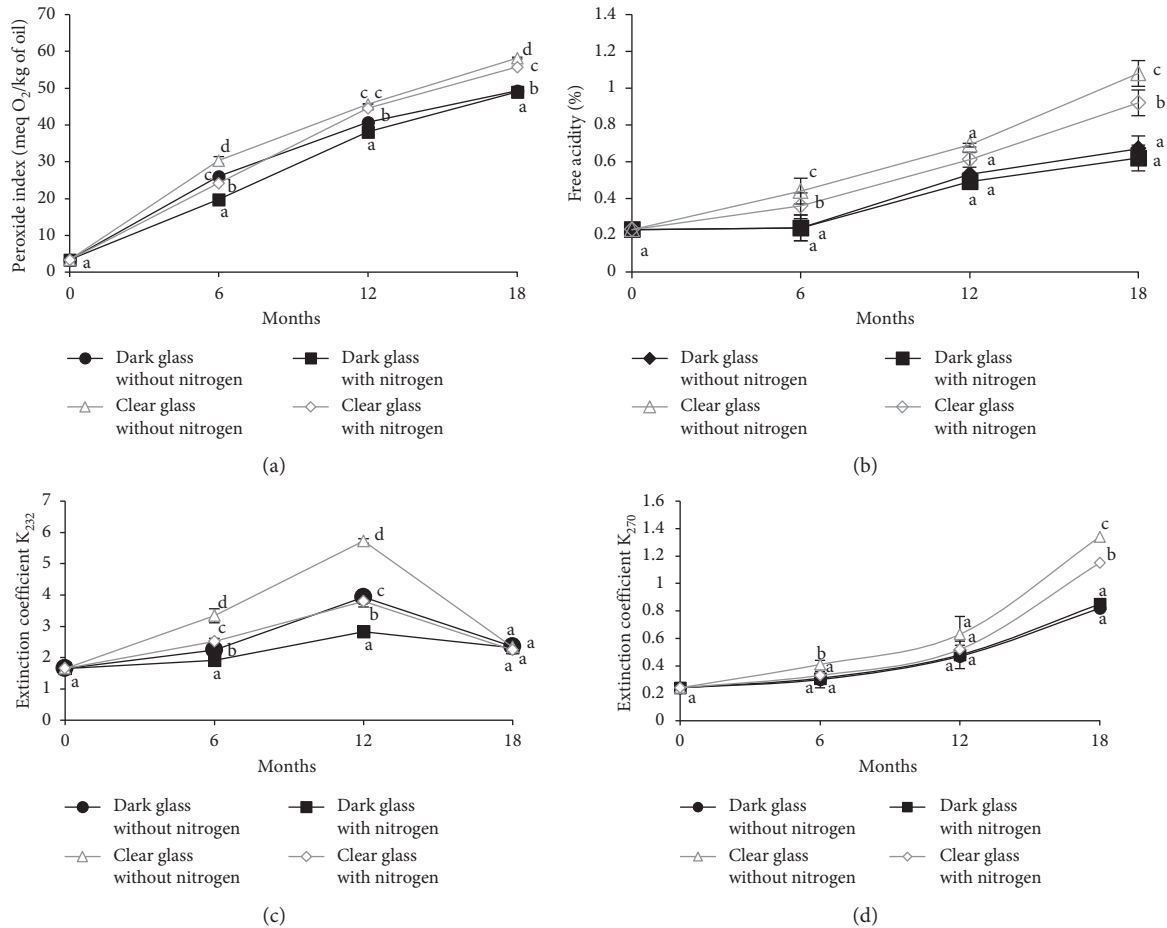


FIGURE 2: Evolution of quality parameters of prickly pear seeds oil during storage at various packaging conditions: dark glass bottles/clear glass bottles, with nitrogen/without nitrogen. (a) Peroxide index. (b) Free acidity. (c) Specific extinction coefficient  $K_{232}$ . (d) Specific extinction coefficient  $K_{270}$ . The error bars represent the standard deviation of three replicates. Means with the same letter for the same storage time are not significantly different according to Student–Newman–Keuls test at  $P < 0.05$ .

night) to 40°C (indoor hottest day). The monitoring lipid oxidation of PPSO stored at uncontrolled room temperature showed the intense increase of peroxide index and conjugated dienes ( $K_{232}$ ) after 6 and 12 months of storage. The values varied from 3.40 to 38.80 meq  $O_2$ /kg of oil and from 1.65 to 3.93, respectively (Figures 1(a) and 1(c)). The formation of secondary oxidation products was more significant after 12 months of storage;  $K_{270}$  has increased from 0.24 to 0.47.

At 18 months of storage, the formation of these secondary oxidation products in PPSO was pronounced but significantly different from that recorded for PPSO stored at 40°C; the  $K_{270}$  values were 0.82 and 0.99, respectively (Figure 1(d)). For free acidity parameter, the increase was detected between 6 and 18 months of storage to reach a very close level to that of PPSO stored at 40°C during 18 months (0.67% for PPSO at uncontrolled temperature versus 0.72% for PPSO at 40°C) (Figure 1(b)). Overall, results showed that the oxidative stability of PPSO stored at uncontrolled temperature was severely affected by the fluctuation of temperature after 12 months of storage. Nevertheless, fatty acids composition of PPSO was not affected.

**3.2. Effect of Packaging Conditions on PPSO Quality during Storage.** The results of the two-way ANOVA, with the objective being the study of the effect of time and packaging interaction on the PPSO quality during storage, have shown, as for time and temperature factors presented above, that all the studied quality parameters (peroxide index, free acidity, and specific extinction coefficients  $K_{232}$  and  $K_{270}$ ) have showed very high significance difference ( $P < 0.001$ ) (Figure 2). Likewise, fatty acids composition has shown low variability. The interaction “time \* packaging conditions” was very highly significant ( $P < 0.001$ ) for only the three fatty acids C18:0, C18:1, and C18:2 (Table 2). The effect of each packaging condition on the PPSO quality during storage is described below.

**3.2.1. Effect of Bubbling Oil with Nitrogen Gas.** Bubbling oil with nitrogen gas impacted hydroperoxide formation in PPSO especially during 6 and 12 months of storage. The presence of nitrogen, as conditioner gas, contributed to reducing the peroxidation of oils during 6 months both in dark glass and clear glass bottles. The peroxide index

TABLE 2: Fatty acids composition of prickly pear seeds oil as a function of storage time at different packaging conditions\*.

Packaging conditions	Time** (months)	Fatty acids (%)									
		Myristic acid C14:0	Palmitic acid C16:0	Palmitoleic acid C16:1	Stearic acid C18:0	Oleic acid C18:1	Linoleic acid C18:2	Linolenic acid C18:3	Arachic acid C20:0	Eicosenoic acid C20:1	
		0.07 <sup>a</sup> ± 0.04	12.34 <sup>a</sup> ± 0.01	0.74 <sup>a</sup> ± 0.03	3.24 <sup>a</sup> ± 0.05	21.25 <sup>a</sup> ± 0.10	61.44 <sup>a</sup> ± 0.06	0.24 <sup>a</sup> ± 0.07	0.31 <sup>a</sup> ± 0.01	0.29 <sup>a</sup> ± 0.07	
Dark glass bottles without nitrogen	6	0.05 <sup>a</sup> ± 0.02	12.34 <sup>a</sup> ± 0.01	0.75 <sup>a</sup> ± 0.03	3.24 <sup>a</sup> ± 0.05	21.25 <sup>a</sup> ± 0.10	61.42 <sup>a</sup> ± 0.05	0.26 <sup>a</sup> ± 0.01	0.29 <sup>a</sup> ± 0.02	0.33 <sup>a</sup> ± 0.05	
	12	0.05 <sup>a</sup> ± 0.02	12.29 <sup>a</sup> ± 0.02	0.74 <sup>a</sup> ± 0.00	3.29 <sup>a</sup> ± 0.06	21.27 <sup>a</sup> ± 0.07	61.56 <sup>b</sup> ± 0.12	0.26 <sup>a</sup> ± 0.03	0.29 <sup>a</sup> ± 0.01	0.27 <sup>a</sup> ± 0.02	
	18	0.07 <sup>a</sup> ± 0.03	12.28 <sup>a</sup> ± 0.04	0.75 <sup>a</sup> ± 0.00	3.34 <sup>a</sup> ± 0.08	21.28 <sup>a</sup> ± 0.03	61.42 <sup>c</sup> ± 0.12	0.23 <sup>b</sup> ± 0.04	0.30 <sup>a</sup> ± 0.01	0.28 <sup>a</sup> ± 0.05	
Dark glass bottles with nitrogen	6	0.05 <sup>a</sup> ± 0.03	12.33 <sup>a</sup> ± 0.02	0.76 <sup>a</sup> ± 0.02	3.27 <sup>a</sup> ± 0.03	21.24 <sup>a</sup> ± 0.14	61.45 <sup>a</sup> ± 0.05	0.25 <sup>a</sup> ± 0.09	0.29 <sup>a</sup> ± 0.01	0.32 <sup>a</sup> ± 0.03	
	12	0.08 <sup>a</sup> ± 0.03	12.27 <sup>a</sup> ± 0.10	0.73 <sup>a</sup> ± 0.03	3.32 <sup>a</sup> ± 0.02	21.26 <sup>a</sup> ± 0.02	61.30 <sup>ab</sup> ± 0.05	0.27 <sup>b</sup> ± 0.01	0.34 <sup>a</sup> ± 0.03	0.32 <sup>a</sup> ± 0.00	
	18	0.07 <sup>a</sup> ± 0.01	12.25 <sup>a</sup> ± 0.10	0.73 <sup>a</sup> ± 0.03	3.30 <sup>a</sup> ± 0.01	21.30 <sup>a</sup> ± 0.08	61.48 <sup>c</sup> ± 0.11	0.24 <sup>b</sup> ± 0.06	0.27 <sup>a</sup> ± 0.09	0.30 <sup>a</sup> ± 0.04	
Clear glass bottles without nitrogen	6	0.06 <sup>a</sup> ± 0.04	12.36 <sup>a</sup> ± 0.07	0.75 <sup>a</sup> ± 0.01	3.31 <sup>a</sup> ± 0.05	21.29 <sup>a</sup> ± 0.10	61.26 <sup>a</sup> ± 0.05	0.25 <sup>a</sup> ± 0.04	0.32 <sup>a</sup> ± 0.04	0.34 <sup>a</sup> ± 0.03	
	12	0.08 <sup>a</sup> ± 0.02	12.36 <sup>a</sup> ± 0.11	0.74 <sup>a</sup> ± 0.04	3.30 <sup>a</sup> ± 0.06	21.40 <sup>a</sup> ± 0.07	61.15 <sup>a</sup> ± 0.02	0.24 <sup>a</sup> ± 0.01	0.32 <sup>a</sup> ± 0.06	0.26 <sup>a</sup> ± 0.05	
	18	0.06 <sup>a</sup> ± 0.04	12.35 <sup>a</sup> ± 0.08	0.75 <sup>a</sup> ± 0.04	3.71 <sup>b</sup> ± 0.02	23.57 <sup>c</sup> ± 0.03	58.99 <sup>a</sup> ± 0.14	0.16 <sup>b</sup> ± 0.06	0.26 <sup>a</sup> ± 0.01	0.26 <sup>a</sup> ± 0.05	
Clear glass bottles with nitrogen	6	0.06 <sup>a</sup> ± 0.02	12.32 <sup>a</sup> ± 0.06	0.76 <sup>a</sup> ± 0.04	3.26 <sup>a</sup> ± 0.05	21.17 <sup>a</sup> ± 0.06	61.42 <sup>a</sup> ± 0.11	0.24 <sup>a</sup> ± 0.02	0.31 <sup>a</sup> ± 0.01	0.31 <sup>a</sup> ± 0.02	
	12	0.06 <sup>a</sup> ± 0.02	12.31 <sup>a</sup> ± 0.00	0.69 <sup>a</sup> ± 0.03	3.31 <sup>a</sup> ± 0.02	21.30 <sup>a</sup> ± 0.09	61.37 <sup>ab</sup> ± 0.07	0.32 <sup>a</sup> ± 0.11	0.28 <sup>a</sup> ± 0.05	0.24 <sup>a</sup> ± 0.05	
	18	0.04 <sup>a</sup> ± 0.02	12.25 <sup>a</sup> ± 0.02	0.73 <sup>a</sup> ± 0.02	3.51 <sup>a</sup> ± 0.08	21.74 <sup>b</sup> ± 0.03	61.03 <sup>b</sup> ± 0.01	0.27 <sup>b</sup> ± 0.01	0.20 <sup>a</sup> ± 0.07	0.27 <sup>a</sup> ± 0.01	

\* Means with the same letter within the column for the same storage time are not significantly different according to Student–Newman–Keuls test at  $P < 0.05$ . \*\* Time 0 corresponds to the beginning of the trial ( $T_0$ ).

increased significantly after 6 months from 3.40 to 26.00 meq O<sub>2</sub>/kg of oil in the absence of nitrogen and from 3.40 to 19.78 meq O<sub>2</sub>/kg of oil in the presence of nitrogen (Figure 2(a)). The production of conjugated dienes has increased during the 12 months of storage and was affected clearly by nitrogen addition; K<sub>232</sub> values for oil stored in dark glass bottles with nitrogen and without nitrogen were 2.83 and 3.93, respectively, after 12 months of storage. However, at 18 months, all oils presented the same K<sub>232</sub> values (Figure 2(c)). The effect of nitrogen on free acidity and the production of secondary oxidation products (K<sub>270</sub>) of PPSO was only depicted for oil stored in clear glass bottles (Figures 2(b) and 2(d)).

**3.2.2. Effect of Light Protection.** The protection of PPSO from sunlight was essential to limit hydroperoxides formation in PPSO during 18 months. At 12 months, peroxide index has increased from 3.40 to 45.72 meq O<sub>2</sub>/kg of oil (for clear glass bottles) and from 3.40 to 38.09 meq O<sub>2</sub>/kg of oil (for dark glass bottles) (Figure 2(a)). A significant difference of K<sub>232</sub> was also depicted between oils stored in dark glass bottles and clear glass bottles during the same period. The values have increased from 1.65 to 3.34 and from 1.65 to 5.73 for oils protected and unprotected from sunlight respectively. At 18 months of storage, oils presented the same ratio of primary products; K<sub>232</sub> decreased and no significant difference was noticed between the different storage conditions (Figure 2(c)). It is probably due to the fact that at 18 months, the primary oxidation products were quickly converted to secondary oxidation products.

The combined action of sunlight and oxygen availability affected after 12 months the secondary oxidation rate of oil stored in clear glass bottles without nitrogen. It seems that the contribution of oxygen in PPSO oxidative process is only significant when this oil is exposed to sunlight for a long time (Figures 2(c) and 2(d)). The percentages of free acidity were 1.08% and 0.67% for oils stored in clear glass and dark glass bottles, respectively (Figure 2(b)), indicating that the light is a major factor for the induction of triacylglycerol oxidation in PPSO. A similar impact of light on triacylglycerol degradation has been reported for argan oil [29].

Concerning the impact of sunlight exposition and oxygen availability on the composition of fatty acids, the changes occurred only for oil stored in clear glass bottles without nitrogen after 12 months. Linoleic acid (C18:2) decreased from 61.44% to 58.99% but oleic acid (C18:1) increased from 21.25% to 23.57% (Table 2). These results did not agree with the findings reported for argan oil. The composition in fatty acids did not change during 24 months of storage in both types of bottles (dark and clear) [29]. Thus, for a long period of storage, it is necessary to protect the PPSO from oxygen and light and more precaution should be given to the packaging material.

**3.3. Detection of PPSO Adulteration.** As indicated above in the oxidative stability test, the quality parameters of pure PPSO freshly extracted in particular free acidity, peroxide value, and K<sub>232</sub> and K<sub>270</sub> specific extinction coefficients are

(0.24 ± 0.01 % of oleic acid), (3.40 ± 0.20 meq of O<sub>2</sub>/kg of oil), and (1.64 ± 0.05) and (0.27 ± 0.02), respectively. The L\*a\*b\* color parameters ((77.96 ± 0.72), (-1.19 ± 0.02), and (19.58 ± 0.15), respectively) show that this oil has yellow-green color with high content of α-pheophytin green stable pigment (3.02 ± 0.06 ppm) although its chlorophyll content is relatively low (0.99 ± 0.01 ppm). In this work, the PPSO fatty acids profile was especially used as markers for detecting adulteration. In fact, fatty acids compositions of pure and mixed oils have allowed identifying the oil levels of PPSO adulteration. These levels depended on the nature of fatty acids (Tables 3 and 4).

For mixtures with olive oil, the identified level to detect PPSO adulteration was 15%. Indeed, the significant increase of oleic acid from 20.17% in pure PPSO to 30.42% in adulterated PPSO at 15% can be considered as a marker of adulteration (Table 3). The percentage of this acid did not exceed 24.3% in reported fatty acids profiles of pure PPSO [2, 11]. Concerning palmitic acid and linoleic acid, even if their respective contents (11.82% and 52.74%) in 15%-olive oil-adulterated-PPSO differ significantly from their contents in pure PPSO oil (12.15% and 60.21%), they cannot be considered as markers of PPSO adulteration, since obtained values fall in the range of values reported in literature for pure PPSO [2, 9, 11]. For mixtures with almond oil, and up to 15%, the same marker (oleic acid) can be used. The percentage of oleic acid has increased from 20.17% in pure PPSO to 28.66% in the 15%-almond oil-adulterated PPSO (Table 3). Under this rate, we cannot detect any adulteration of PPSO with almond oil using fatty acids profile.

Mixing PPSO with rapeseed oil at different rates (1%, 5%, 10%, 15%, and 20%) showed that linolenic acid, essential fatty acid from the omega 3 family that cannot be synthesized by the body, can be used as a marker to detect the adulteration with this oil (Table 4) in which the content of this fatty acid (3.98%) exceeded those reported for pure PPSO [2, 9, 11]. Therefore, the detection level of the adulteration with rapeseed oil is 20% which is higher than that of the adulteration with olive or almond oils (15%).

For all mixtures carried out using sunflower oil at different rates including the higher rate of 50%, the occurring changes in fatty acids distribution, even if they were significant (Table 4), never exceeded the values reported for pure PPSO [2, 9, 11]. Consequently, fatty acids profile cannot be used for the detection of suspected adulteration with sunflower oil. Other minor compounds including sterols and tocopherols should be investigated to depict the adulteration with this oil.

Considering the iodine value (IV) which is an indicator of the oxidative stability of the oil, the pure PPSO showed to be less stable than olive and almond oils by presenting the highest IV (129.13). However, PPSO is more stable than colza and sunflower oils (having 154.20 and 137.83 for IV, respectively). The IV recorded in this study is higher than that reported by De Wit et al. [30] for PPSO of five cultivars of the same species (*Opuntia ficus-indica*) (IV ranged from 110.68 to 124.24). Regarding the adulteration, significant differences have been demonstrated between IV of pure PPSO and oil mixtures: (PPSO and olive oil), (PPSO and



TABLE 3: Comparison of fatty acids composition of pure prickly pear seeds oil (PPOS) and PPSO mixed at different rates with (A) olive oil and almond oil.

Fatty acids (%)	Pure oils													
	PPOS			Almond oil			Olive oil			Almond oil/PPSO				
	100%	100%	100%	100%	100%	100%	1%	5%	10%	15%	1%	5%	10%	15%
Myristic acid (C14:0)	0.04 <sup>a</sup> ± 0.00	0.05 <sup>a</sup> ± 0.00	0.05 <sup>a</sup> ± 0.00	0.00 <sup>a</sup> ± 0.01	0.07 <sup>a</sup> ± 0.00	0.04 <sup>a</sup> ± 0.00	0.03 <sup>a</sup> ± 0.01	0.04 <sup>a</sup> ± 0.00	0.05 <sup>a</sup> ± 0.01	0.03 <sup>a</sup> ± 0.01	0.04 <sup>a</sup> ± 0.00	0.00 <sup>a</sup> ± 0.00	0.00 <sup>a</sup> ± 0.00	0.00 <sup>a</sup> ± 0.01
Palmitic acid (C16:0)	12.15 <sup>c</sup> ± 0.04	8.80 <sup>b</sup> ± 0.11	8.80 <sup>b</sup> ± 0.11	4.44 <sup>a</sup> ± 0.04	12.36 <sup>c</sup> ± 0.24	12.18 <sup>c</sup> ± 0.00	11.82 <sup>d</sup> ± 0.07	12.15 <sup>c</sup> ± 0.04	12.11 <sup>c</sup> ± 0.00	11.82 <sup>d</sup> ± 0.07	12.15 <sup>c</sup> ± 0.04	12.45 <sup>c</sup> ± 0.03	11.54 <sup>d</sup> ± 0.20	11.02 <sup>c</sup> ± 0.07
Palmitoleic acid (C16:1)	0.52 <sup>a</sup> ± 0.06	0.40 <sup>a</sup> ± 0.01	0.40 <sup>a</sup> ± 0.01	1.32 <sup>b</sup> ± 0.04	0.56 <sup>a</sup> ± 0.02	0.52 <sup>a</sup> ± 0.14	0.56 <sup>a</sup> ± 0.00	0.52 <sup>a</sup> ± 0.06	0.53 <sup>a</sup> ± 0.13	0.56 <sup>a</sup> ± 0.00	0.52 <sup>a</sup> ± 0.06	0.62 <sup>a</sup> ± 0.03	0.58 <sup>a</sup> ± 0.01	0.50 <sup>a</sup> ± 0.04
Stearic acid (C18:0)	3.88 <sup>d</sup> ± 0.06	1.85 <sup>a</sup> ± 0.15	1.85 <sup>a</sup> ± 0.15	2.23 <sup>b</sup> ± 1.02	3.22 <sup>c</sup> ± 0.34	3.26 <sup>c</sup> ± 0.13	3.08 <sup>c</sup> ± 0.12	3.80 <sup>d</sup> ± 0.06	3.06 <sup>c</sup> ± 0.06	3.08 <sup>c</sup> ± 0.12	3.80 <sup>d</sup> ± 0.06	3.03 <sup>c</sup> ± 0.23	3.15 <sup>c</sup> ± 0.11	3.09 <sup>c</sup> ± 0.00
Oleic acid (C18:1)	20.17 <sup>a</sup> ± 0.11	74.11 <sup>f</sup> ± 0.80	74.11 <sup>f</sup> ± 0.80	71.37 <sup>f</sup> ± 0.02	21.99 <sup>b</sup> ± 1.82	23.61 <sup>c</sup> ± 0.31	30.42 <sup>e</sup> ± 0.03	20.17 <sup>a</sup> ± 0.11	24.15 <sup>c</sup> ± 0.21	30.42 <sup>e</sup> ± 0.03	20.17 <sup>a</sup> ± 0.11	20.36 <sup>a</sup> ± 0.03	25.46 <sup>c</sup> ± 0.87	28.66 <sup>e</sup> ± 0.13
Linoleic acid (C18:2)	60.21 <sup>d</sup> ± 0.05	12.72 <sup>a</sup> ± 0.01	12.72 <sup>a</sup> ± 0.01	20.51 <sup>b</sup> ± 0.10	61.05 <sup>d</sup> ± 2.50	59.77 <sup>d</sup> ± 0.02	52.74 <sup>c</sup> ± 0.09	60.20 <sup>d</sup> ± 0.05	59.30 <sup>d</sup> ± 0.02	52.74 <sup>c</sup> ± 0.09	60.20 <sup>d</sup> ± 0.05	61.78 <sup>e</sup> ± 0.64	59.17 <sup>c</sup> ± 0.72	55.6 <sup>c</sup> ± 0.22
Linolenic acid (C18:3)	0.35 <sup>a</sup> ± 0.03	1.00 <sup>b</sup> ± 0.07	1.00 <sup>b</sup> ± 0.07	0.03 <sup>a</sup> ± 0.00	0.22 <sup>a</sup> ± 0.04	0.15 <sup>a</sup> ± 0.05	0.35 <sup>a</sup> ± 0.05	0.35 <sup>a</sup> ± 0.03	0.24 <sup>a</sup> ± 0.04	0.35 <sup>a</sup> ± 0.05	0.35 <sup>a</sup> ± 0.03	0.47 <sup>a</sup> ± 0.13	0.37 <sup>a</sup> ± 0.06	0.21 <sup>a</sup> ± 0.01
Arachic acid (C20:0)	0.26 <sup>b</sup> ± 0.01	0.27 <sup>b</sup> ± 0.00	0.27 <sup>b</sup> ± 0.00	0.23 <sup>b</sup> ± 0.06	0.35 <sup>b</sup> ± 0.02	0.34 <sup>b</sup> ± 0.07	0.21 <sup>b</sup> ± 0.03	0.26 <sup>b</sup> ± 0.01	0.15 <sup>a</sup> ± 0.02	0.21 <sup>b</sup> ± 0.03	0.26 <sup>b</sup> ± 0.01	0.27 <sup>b</sup> ± 0.02	0.14 <sup>a</sup> ± 0.03	0.34 <sup>b</sup> ± 0.01
Eicosenoic acid (C20:1)	0.20 <sup>a</sup> ± 0.04	0.28 <sup>b</sup> ± 0.02	0.28 <sup>b</sup> ± 0.02	0.24 <sup>a</sup> ± 0.05	0.22 <sup>a</sup> ± 0.02	0.32 <sup>b</sup> ± 0.01	0.28 <sup>b</sup> ± 0.01	0.20 <sup>a</sup> ± 0.01	0.29 <sup>b</sup> ± 0.10	0.28 <sup>b</sup> ± 0.01	0.20 <sup>a</sup> ± 0.01	0.18 <sup>a</sup> ± 0.02	0.16 <sup>a</sup> ± 0.01	0.29 <sup>b</sup> ± 0.00
Iodine value	129.13 <sup>c</sup> ± 0.80	92.90 <sup>a</sup> ± 0.14	92.90 <sup>a</sup> ± 0.14	102.17 <sup>b</sup> ± 0.25	131.98 <sup>e</sup> ± 0.12	130.79 <sup>f</sup> ± 0.21	124.80 <sup>c</sup> ± 0.19	129.12 <sup>c</sup> ± 0.72	130.76 <sup>f</sup> ± 0.10	124.80 <sup>c</sup> ± 0.19	129.12 <sup>c</sup> ± 0.72	132.59 <sup>i</sup> ± 0.22	132.11 <sup>h</sup> ± 0.36	127.98 <sup>d</sup> ± 0.15

\*Values having same letter within the line are not significantly different according to Student-Newman-Keuls test at  $P < 0.05$ .

TABLE 4: Comparison of fatty acids composition of pure prickly pear seeds oil (PPOS) and PPSO mixed at different rates with (B) rapeseed oil and sunflower oil\*.

Fatty acids (%)	Pure oils										Mixture rates (%)																	
	PPSO		Rapeseed oil		Sunflower oil		Rapeseed oil/PPSO		Sunflower oil/PPSO		1%		15%		20%		5%		10%		20%		30%		50%			
	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%		
Myristic acid (C14:0)	0.04 <sup>a</sup> ± 0.00	0.02 <sup>a</sup> ± 0.00	0.04 <sup>a</sup> ± 0.00	0.02 <sup>a</sup> ± 0.00	0.04 <sup>a</sup> ± 0.00	0.02 <sup>a</sup> ± 0.00	0.04 <sup>a</sup> ± 0.00	0.02 <sup>a</sup> ± 0.00	0.04 <sup>a</sup> ± 0.00	0.02 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	0.08 <sup>a</sup> ± 0.00	
Palmitic acid (C16:0)	124.5 <sup>f</sup> ± 0.04	4.80 <sup>b</sup> ± 0.05	6.66 <sup>b</sup> ± 0.03	12.30 <sup>f</sup> ± 0.20	11.46 <sup>d</sup> ± 0.05	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.28 <sup>d</sup> ± 0.05	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04	11.77 <sup>e</sup> ± 0.23	9.84 <sup>e</sup> ± 0.04
Palmitoleic acid (C16:1)	0.52 <sup>b</sup> ± 0.06	0.66 <sup>b</sup> ± 0.00	0.05 <sup>a</sup> ± 0.01	0.50 <sup>b</sup> ± 0.05	0.45 <sup>b</sup> ± 0.03	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.40 <sup>b</sup> ± 0.01	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02	0.48 <sup>b</sup> ± 0.02	0.47 <sup>b</sup> ± 0.02
Stearic acid (C18:0)	3.88 <sup>c</sup> ± 0.06	1.79 <sup>b</sup> ± 0.11	3.62 <sup>c</sup> ± 0.16	3.27 <sup>c</sup> ± 0.14	3.36 <sup>c</sup> ± 0.12	3.15 <sup>bc</sup> ± 0.02	2.79 <sup>b</sup> ± 0.06	3.13 <sup>bc</sup> ± 0.02	3.36 <sup>c</sup> ± 0.12	2.79 <sup>b</sup> ± 0.06	3.15 <sup>bc</sup> ± 0.02	2.79 <sup>b</sup> ± 0.06	3.13 <sup>bc</sup> ± 0.02	3.36 <sup>c</sup> ± 0.12	2.79 <sup>b</sup> ± 0.06	3.15 <sup>bc</sup> ± 0.02	2.79 <sup>b</sup> ± 0.06	3.13 <sup>bc</sup> ± 0.02	3.36 <sup>c</sup> ± 0.12	2.79 <sup>b</sup> ± 0.06	3.15 <sup>bc</sup> ± 0.02	2.79 <sup>b</sup> ± 0.06	3.13 <sup>bc</sup> ± 0.02	3.36 <sup>c</sup> ± 0.12	2.79 <sup>b</sup> ± 0.06	3.15 <sup>bc</sup> ± 0.02	2.79 <sup>b</sup> ± 0.06	3.13 <sup>bc</sup> ± 0.02
Oleic acid (C18:1)	201.7 <sup>a</sup> ± 0.11	22.29 <sup>b</sup> ± 0.21	26.28 <sup>c</sup> ± 0.03	22.30 <sup>b</sup> ± 1.80	23.96 <sup>c</sup> ± 0.19	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	21.00 <sup>a</sup> ± 0.14	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	21.00 <sup>a</sup> ± 0.14	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	21.00 <sup>a</sup> ± 0.14	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	21.00 <sup>a</sup> ± 0.14	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	24.36 <sup>d</sup> ± 0.11	32.94 <sup>d</sup> ± 0.03	24.36 <sup>d</sup> ± 0.11
Linoleic acid (C18:2)	60.21 <sup>b</sup> ± 0.05	59.77 <sup>b</sup> ± 0.71	62.28 <sup>c</sup> ± 0.26	59.97 <sup>b</sup> ± 2.32	58.27 <sup>b</sup> ± 0.04	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	60.88 <sup>b</sup> ± 0.02	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	60.88 <sup>b</sup> ± 0.02	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	60.88 <sup>b</sup> ± 0.02	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	60.88 <sup>b</sup> ± 0.02	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	57.48 <sup>b</sup> ± 0.25	49.23 <sup>a</sup> ± 0.46	57.48 <sup>b</sup> ± 0.25
Linolenic acid (C18:3)	0.35 <sup>b</sup> ± 0.03	9.13 <sup>b</sup> ± 0.10	0.32 <sup>b</sup> ± 0.14	0.35 <sup>b</sup> ± 0.08	1.50 <sup>a</sup> ± 0.03	1.51 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.51 <sup>a</sup> ± 0.01	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.51 <sup>a</sup> ± 0.01	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.51 <sup>a</sup> ± 0.01	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.51 <sup>a</sup> ± 0.01	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.70 <sup>a</sup> ± 0.01	3.98 <sup>a</sup> ± 0.05	1.70 <sup>a</sup> ± 0.01
Arachic acid (C20:0)	0.26 <sup>a</sup> ± 0.01	0.40 <sup>b</sup> ± 0.06	0.20 <sup>a</sup> ± 0.10	0.22 <sup>a</sup> ± 0.11	0.30 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.02	0.29 <sup>a</sup> ± 0.01	0.25 <sup>a</sup> ± 0.02
Eicosenic acid (C20:1)	0.20 <sup>a</sup> ± 0.01	0.32 <sup>a</sup> ± 0.01	0.10 <sup>a</sup> ± 0.04	0.40 <sup>b</sup> ± 0.00	0.41 <sup>b</sup> ± 0.15	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01	0.37 <sup>b</sup> ± 0.01	0.40 <sup>b</sup> ± 0.01
Iodine value	129.13 <sup>a</sup> ± 0.80	154.201 ± 0.10	137.83 <sup>b</sup> ± 0.10	130.59 <sup>b</sup> ± 0.21	132.03 <sup>c</sup> ± 0.16	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11	130.58 <sup>b</sup> ± 0.14	134.15 <sup>c</sup> ± 0.11

\*Values having the same letter within the line are not significantly different according to Student-Newman-Keuls test at  $P < 0.05$ .

almond oil), (PPSO and rapeseed oil), and (PPSO and sunflower oil) (Tables 3 and 4) (ranging from 124.80 to 136.10). The variation of IV as a function of oil adulteration rates shows that IV could be an indicator of the presence of sunflower oil detectable from 1%. Mixing the sunflower oil with PPSO resulted in a significant increase of the IV with the increase of the sunflower oil rate in the mixture. This could be explained by the fact that the sunflower oil is the richest in linoleic acid (C18:2) compared to other studied oils.

#### 4. Conclusions

In this work, two aspects relating to the PPSO preservation and adulteration of an immense importance for sanitary and commercial quality were studied. It has shown that PPSO stored at 4°C for 18 months preserve the initial quality based on the evolution of quality parameters and fatty acid compositions. However, at 40°C, an intense lipid oxidative process occurred after 6 months of storage. The changes have also affected fatty acids composition especially rates of linoleic and oleic acids. The shelf-life of oils stored at 25°C and at uncontrolled room temperature can be limited to 6 months. The PPSO bubbling with nitrogen has improved its stability when it has been stored in clear glass bottles. Thus, it is recommended to protect PPSO from oxygen and light by using dark glass bottles for a long period of storage even if the use of this packaging material is not easily accepted by some consumers. The levels of adulteration detection using fatty acids as markers are relatively high. The detection of oil adulteration can be depicted up to 15% of olive and almond oils and up to 20% of rapeseed oil. The iodine value could also be an indicator of the presence of sunflower oil detectable in PPSO from 1%. Therefore, other minor compounds including sterols and tocopherols should be investigated to depict the adulteration of PPSO with cheaper oils and to determine lower levels of detection for the authentication of PPSO purity.

#### Data Availability

The data used to support the findings of this study are available from the first author Fatima Ettalibi (fa.ettalibi@gmail.com) upon request.

#### Disclosure

This research was performed at the Regional Center for Agricultural Research in Marrakesh belonging to the National Institute for Agricultural Research (INRA Morocco) in the framework of the Megaproject: "Preservation and Development of the Cactus Sector" of INRA Medium-Term Research Program (PRMT 2017–2020).

#### Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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#### References

- [1] Z. Ghazi, M. Ramdani, M. L. Fauconnier, B. El Mahi, and R. Cheikh, "Fatty acids sterols and vitamin E composition of seed oil of *Opuntia Ficus indica* and *Opuntia dillenii* from Morocco," *Journal of Materials and Environmental Science*, vol. 4, no. 6, pp. 967–972, 2013.
- [2] N. Chougui, A. Tamendjari, W. Hamidj et al., "Oil composition and characterisation of phenolic compounds of *Opuntia ficus-indica* seeds," *Food Chemistry*, vol. 139, no. 1–4, pp. 796–803, 2013.
- [3] M. F. Ramadan and J.-T. Mörsel, "Oil cactus pear (*Opuntia ficus-indica* L.)," *Food Chemistry*, vol. 82, no. 3, pp. 339–345, 2003.
- [4] S. Zine, S. Gharby, and M. El Hadek, "Physicochemical characterization of *Opuntia ficus-indica* seed oil from Morocco," *Biosciences Biotechnology Research Asia*, vol. 10, pp. 1–7, 2013.
- [5] B. Matthäus and M. M. Özcan, "Habitat effects on yield, fatty acid composition and tocopherol contents of prickly pear (*Opuntia ficus-indica* L.) seed oils," *Scientia Horticulturae*, vol. 131, pp. 95–98, 2012.
- [6] F. Taoufik, S. Zine, M. El Hadek et al., "Oil content and main constituents of cactus seed oils *Opuntia Ficus Indica* of different origin in Morocco," *Mediterranean Journal of Nutrition and Metabolism*, vol. 8, no. 2, pp. 85–92, 2015.
- [7] B. S. Chu, B. S. Baharin, and S. Y. Quek, "Factors affecting pre-concentration of tocopherols and tocotrienols from palm fatty acid distillate by lipase-catalysed hydrolysis," *Journal of Food Chemistry*, vol. 79, no. 1, pp. 9–55, 2002.
- [8] F. Saloua, N. I. Eddine, and Z. Hedi, "Chemical composition and profile characteristics of Osage orange *Maclura pomifera* (Rafin.) Schneider seed and seed oil," *Industrial Crops and Products*, vol. 29, no. 1, pp. 1–8, 2009.
- [9] M. Ennouri, H. Fetoui, E. Bourret, N. Zeghal, and H. Attia, "Evaluation of some biological parameters of *Opuntia ficus indica*. 1. Influence of a seed oil supplemented diet on rats," *Bioresource Technology*, vol. 97, no. 12, pp. 1382–1386, 2006.
- [10] D. B. Hmamou, R. Salghi, A. Zarrouk et al., "Corrosion inhibition of steel in 1M hydrochloric acid medium by chamomile essential oils," *International Journal of Electrochemical Science*, vol. 7, pp. 2361–2373, 2012.
- [11] B. Matthäus, D. Guillaume, S. Gharby, A. Haddad, H. Harhar, and Z. Charrouf, "Effect of processing on the quality of edible argan oil," *Food Chemistry*, vol. 120, no. 2, pp. 426–432, 2010.
- [12] N. Abel, B. T. Rotabakk, T. Rustad, and J. Lerfall, "The influence of lipid composition, storage temperature, and modified atmospheric gas combinations on the solubility of CO<sub>2</sub> in a seafood model product," *Journal of Food Engineering*, vol. 216, pp. 151–158, 2018.

- [13] Y. B. C. Man, J. L. Liu, B. Jamilah, and R. A. Rahman, "Quality changes of refined-bleached-deodorized (rbd) palm olein, soybean oil and their blends during deep-fat frying," *Journal of Food Lipids*, vol. 6, no. 3, pp. 181–193, 1999.
- [14] N. Vanstone, A. Moore, P. Martos, and S. Neethirajan, "Detection of the adulteration of extra virgin olive oil by near-infrared spectroscopy and chemometric techniques," *Food Quality and Safety*, vol. 2, no. 4, pp. 189–198, 2018.
- [15] A. H. El-Hamdy and N. K. El-Fizga, "Detection of olive oil adulteration by measuring its authenticity factor using reversed-phase high-performance liquid chromatography," *Journal of Chromatography A*, vol. 708, no. 2, pp. 351–355, 1995.
- [16] S. Mildner-Szkudlarz and H. H. Jeleń, "The potential of different techniques for volatile compounds analysis coupled with PCA for the detection of the adulteration of olive oil with hazelnut oil," *Food Chemistry*, vol. 110, no. 3, pp. 751–761, 2008.
- [17] AFNOR, *Recueil de Normes Françaises des Corps Gras, Graines Oléagineuses, Produits Dérivés*, Association Française de Normalisation, Paris, France, 3rd edition, 1984.
- [18] IOC, *Spectrophotometric Analysis in Ultraviolet*, International Olive Council, Madrid, Spain, 2010.
- [19] J. P. Wolff, *Manuel d'analyse des corps gras*, Vol. 517, Azoulay, Paris, France, 1968.
- [20] E. Psoyiadou and M. Tsimidou, "Pigments in Greek virgin olive oils: occurrence and levels," *Journal of the Science of Food and Agriculture*, vol. 81, no. 7, pp. 640–647, 2001.
- [21] M. I. Mosquera-Minguez, L. Rejano, B. Guandul, A. H. Sanchez, and J. Garido, "Color pigment, correlation in virgin olive oil," *Journal of the American Oil Chemists' Society*, vol. 68, pp. 332–336, 1991.
- [22] IOC, *Determination of Fatty Acid Methyl Esters by Gas Chromatography*, International Olive Council, Madrid, Spain, 2017.
- [23] H. Duraman and H. Dibeklioğlu, "Characterization of Turkish virgin olive oils produced from early harvest olives," *Journal of the American Oil Chemists' Society*, vol. 86, pp. 663–674, 2009.
- [24] E. R. Sherwin, "Oxidation and antioxidants in fat and oil processing," *Journal of the American Oil Chemists' Society*, vol. 55, no. 11, pp. 809–814, 1978.
- [25] G. Pristouri, A. Badeka, and M. G. Kontominas, "Effect of packaging material headspace, oxygen and light transmission, temperature and storage time on quality characteristics of extra virgin olive oil," *Food Control*, vol. 21, no. 4, pp. 412–418, 2010.
- [26] S. Gómez-Alonso, V. Mancebo-Campos, M. D. Salvador, and G. Fregapane, "Evolution of major and minor components and oxidation indices of virgin olive oil during 21 months storage at room temperature," *Food Chemistry*, vol. 100, no. 1, pp. 36–42, 2007.
- [27] L. Di Giovacchino, M. R. Mucciarella, N. Costantini, M. L. Ferrante, and G. Surricchio, "Use of nitrogen to improve stability of virgin olive oil during storage," *Journal of the American Oil Chemists' Society*, vol. 79, no. 4, pp. 339–344, 2002.
- [28] M. D. Guillén and A. Ruiz, "Study by means of  $^1\text{H}$  nuclear magnetic resonance of the oxidation process undergone by edible oils of different natures submitted to microwave action," *Food Chemistry*, vol. 96, no. 4, pp. 665–674, 2006.
- [29] S. Gharby, H. Harhar, D. Guillaume, A. Haddad, B. Matthäus, and Z. Charrouf, "Oxidative stability of edible argan oil: a two-year study," *LWT—Food Science and Technology*, vol. 44, no. 1, pp. 1–8, 2011.
- [30] M. De Wit, A. Hugo, and N. Shongwe, "Quality assessment of seed oil from selected cactus pear cultivars (*Opuntia ficus-indica* and *Opuntia robusta*)," *Journal of Food Processing and Preservation*, vol. 41, no. 3, Article ID e12898, 2016.