

# Effect of thermal and photo oxidation on the stability of cold-pressed plum and apricot kernel oils

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**Abstract** Oxidative stability of cold-pressed plum (*Prunus domestica*) kernel oil (PKO), and apricot (*Prunus armeniaca*) kernel oil (AKO) were evaluated under thermal-oxidation and photo-oxidation conditions. Changes in peroxide value (PV), conjugated dienes ( $K_{232}$ ), and levels of volatile compounds in the cold-pressed oils were monitored during storage for 12 days under oxidation conditions. Under thermal oxidation conditions, PV values of PKO reached the maximum value (63.8 meq  $O_2$ /kg) after 12 days of storage, while PV values of AKO reached the maximum value (54.5 meq  $O_2$ /kg) after 10 days of storage.  $K_{232}$  values exhibited similar behavior under accelerated oxidation conditions, wherein the highest  $K_{232}$  values were 12.38 and 10.91 for PKO and AKO at the 12th and the 10th day of storage, respectively. Under photo-oxidation conditions, both values recorded similar behavior. At the end of storage (12th day), PV values reached 117.5 and 67.62 meq  $O_2$ /kg for PKO and AKO, respectively. Similarly, the maximum

$K_{232}$  values were 5.72 and 4.56 for PKO and AKO, respectively. Hexanal, and *E*-2-heptenal recorded the values of 149.2 and  $41.83 \times 10^6$  AU for the PKO after 12 days of storage under thermal oxidation conditions, while hexanal, and *E*-2-heptenal reach up to 199.2 and  $58.88 \times 10^6$  AU for the AKO after only 10 days of storage. At the end of 12 days of storage under photo-oxidation of PKO and AKO, the aldehydes were detected as the most identified chemical group (especially hexanal, and *E*-2-heptenal) in the photo-oxidized PKO and AKO.

**Keywords** *Prunus domestica* · *Prunus armeniaca* · Rosaceae · Kernel oil · Agro-industrial by-products · Volatile compounds · Antioxidants

## Abbreviations

AKO	Cold-pressed apricot kernel oil
PKO	Cold-pressed plum kernel oil
PV	Peroxide value
$K_{232}$	Conjugated diene
IUPAC	International Union of Pure and Applied Chemistry
GC	Gas chromatography
FAME	Fatty acid methyl esters
SPME	Solid phase micro extraction
KI	Kovats index

## Introduction

Nowadays, there is an increasing interest on the efficient utilization of agro-industrial by-products. The improper disposal of fruits, vegetables and crops by-products generates pollution problems and a significant loss of biomass that might be used to obtain value-added compounds [1].

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Agro-industrial wastes (i.e., olive pomace, and grape pomace) are a promising raw material for oil production due to their low costs and might be a good alternative for edible oils [2–4]. Non-traditional fats and oils have gained attention due to their useful and health-promoting traits [5–9].

Plums (family Rosaceae) are grown in different Turkish regions [10]. Plums belong to the genus *Prunus* spp. are among the popular processed fruits, with the global production in 2014 reaching 11.2 million tons. In this respect, Turkey is a major plums producer worldwide [11]. During fruit processing tones of fruit pits (agro-industrial by-products) are generated. As the world production of plums continues to rise, utilization of fruit pits will be of importance for the industry and environment [9, 12].

Using the stone fruits (i.e., plum and apricot) as a raw material, the Turkish food industry produces significant amounts of preserved fruits, and juices. Plum (*Prunus domestica*) and apricot (*Prunus armeniaca*), are characterized by high contents of oil, fiber, and phenolics with health-promoting effects [1, 13, 14]. Kernels of apricot and plum are produced as by-products in a significant amount from food industry. Kernels, located inside of the fruit pits, could be considered as a non-traditional source of lipidic bioactive compounds [15–18].

Plum kernels are a good source of oil which can yield over 50% [9], with a high level of oleic acid, and a moderate level of linoleic acid. Plum kernel oil (PKO) contains high amounts of sterols and tocopherols with beneficial traits [19]. Apricot kernels, after removing the stone, are used as roasted, salted tidbit and appetizers [14, 20]. Apricot kernel is a rich source of oil, fiber, and protein [14, 16, 21]. Apricot kernels are used for oil production. Fatty acids are the main constituents (91.5–91.8%) of apricot kernel oil (AKO), including oleic (58.3–73.4%) and linoleic (18.8–31.7%) acid [22].

PKO aroma is rich in benzaldehyde and benzyl alcohol compounds [23]. Benzaldehyde, 2-methyl-propanal, 2-methyl-butyl aldehyde, furfural, nonanal, methylpyrazine, 2,5-dimethyl-pyrazine, methoxy pyrazine, and 3-ethyl-2,5-dimethyl-pyrazine were the major volatile compounds that contribute to AKO aroma [24]. PKO and AKO could be used in cosmetics and personal care products. Besides, PKO and AKO could be used in the food products such as salad dressing and for desert flavoring [25].

Fruit kernel oils contain high levels of bioactive compounds including tocopherols [9, 26], and phenolic compounds [27] which contribute to the stability of the oils. Fruits kernel oils exhibited a strong resistance towards oxidation under the ambient temperature [28], wherein AKO showed high stability under moderate and high temperatures [29]. Recent studies on the stability of oils and fats highlighted the importance of studying lipids stability under thermal and photo-oxidation conditions [30, 31].

Studies on the oxidative stability of cold-pressed PKO and AKO under accelerated oxidative conditions and photo-oxidation conditions have not been carried out up to now. The aim of this work was to investigate the oxidative stability and changes in the volatile compounds of cold-pressed AKO and PKO under thermal and photo-oxidative conditions. The results may open a new and feasible area for the applications of PKO and AKO in the industrial and technological fields.

## Materials and methods

Cold-pressed AKO and PKO were obtained from a local cold pressing factory (Oneva cold pressing, Istanbul, Turkey). High purity chemicals and solvents were commercially provided and used without further purification.

### Fatty acid composition of AKO and PKO

Fatty acid methyl esters (FAME) were prepared according to IUPAC [32]. The gas chromatograph (GC) analysis of FAME was performed using a Shimadzu GC-2010 gas chromatograph equipped with a fused capillary DB-23 fused-silica capillary column (60 m, 0.25 mm i.d., 0.25  $\mu$ m film thickness, Agilent J&W, USA). Helium with a flow rate of 0.70 mL/min was used as a carrier gas. The column temperature was adjusted to be isothermal at 190 °C for 95 min whereas the injector and detector temperatures were 230 and 240 °C, respectively. FAME were quantified as percentages of the total FAME peak areas and identified by the comparison of retention times with the reference standards.

### Thermal oxidation of AKO and PKO

Three grams of oil samples were accurately weighed and placed into a 20-mL brown headspace auto sampler vial capped with a Teflon-lined septum. The oxidation was carried out in a forced-draft air oven at 60 °C. Peroxide value (PV), conjugated diene ( $K_{232}$ ) and volatile compounds released from oil samples were analyzed at the end of every twin-day up to the end of 12th day of storage under accelerated thermal oxidation conditions.

### Photo-oxidation of AKO and PKO

Oil samples (3 g) were precisely poured into 20-mL clear headspace auto sampler vial capped with a Teflon-lined septum, and placed in a lightbox (Test 742, Turkey) equipped with cool white fluorescent lights. The fluorescent radiation intensity was about 3000 lx. Levels of PV,  $K_{232}$ , and volatile compounds in oil samples were measured every 24 h wherein the storage under photo-oxidation conditions was lasted for 12 days.

## Monitoring oxidation parameters

Peroxide values (PV) of the oils were determined iodometrically according to AOCS Cd 8-53 method [33]. The oils were analyzed for conjugated diene ( $K_{232}$ ) according to AOCS Cd 18-90 method [33].

## Analyses of volatile compounds by SPME-GC/MS

Two grams of oil sample were attentively placed in 20 mL headspace screw-top vial and allowed to equilibrate for 15 min at the constant temperature (35 °C). The headspace of samples was extracted for 45 min at 35 °C with the aid of a CTC Combi PAL (CTC Analytics AG, Zwingen, Switzerland) auto sampler with 75  $\mu$ m carboxen/polydimethylsiloxane (CAR/PDMS) solid phase micro extraction (SPME) fiber. Volatile compounds were directly desorbed by inserting the fiber for 10 min into the injection port of GC maintained at the constant temperature (250 °C). Analyses of volatile compounds were performed by means of an Agilent model 7890 Series (Agilent Technologies, Santa Clara, CA, USA) GC in combination with a CTC Combi PAL autosampler and an Agilent 5975 N (Agilent Technologies, Santa Clara, CA, USA) mass selective detector. The compounds were separated in a capillary column of DB-624 (30 m length  $\times$  0.25 mm ID  $\times$  1.4  $\mu$ m film thickness, Agilent Technologies, Santa Clara, CA, USA) with the following temperature program: hold for 5 min at 40 °C; 3 °C/min up to 110 °C; 4 °C/min up to 150 °C; 10 °C/min up to 210 °C and hold for 12 min. The temperatures of the injection port, ion source, quadrupole, and interface were set to be 250, 230, 150, and 240 °C, respectively. Mass spectra were recorded in full scan mode at the electron impact of 70 eV with the scan range from  $m/z$  41 to 400.

Identification of compounds was detected by comparing mass spectra and Kovats index (KI) with the authentic standards and published data, as well as by comparing their mass spectra with the mass spectrometry library of Nist05 (National Institute of Standards and Technology, Gaithersburg MD, USA) and Wiley7.0 (Wiley, NY, USA). The parameters of KI were calculated using the series of *n*-hydrocarbons ( $C_4$  to  $C_{20}$ ). High purity chemicals (hexanal, limonene, *p*-cymene, and nonanal) were purchased from Sigma-Aldrich (St Louis, MO, USA).

## Results and discussion

### Fatty acid composition of PKO and AKO

Fatty acid composition of edible oils varies among plant species and the occurrence of unusual fatty acids can be used for the differentiation of plant families [34]. Fatty acid composition of PKO and AKO is shown in Table 1.

**Table 1** Fatty acid (relative content, %) composition of PKO and AKO

Fatty acid	PKO	AKO
Lauric ( $C_{12:0}$ )	0.03 $\pm$ 0.01	0.02 $\pm$ 0.01
Miristic ( $C_{14:0}$ )	0.01 $\pm$ 0.001	ND
Palmitic ( $C_{16:0}$ )	5.83 $\pm$ 0.04	5.12 $\pm$ 0.05
Palmitoleic ( $C_{16:1}$ )	1.25 $\pm$ 0.02	0.77 $\pm$ 0.03
Stearic ( $C_{18:0}$ )	1.35 $\pm$ 0.01	1.10 $\pm$ 0.02
Oleic ( $C_{18:1}$ )	75.43 $\pm$ 0.09	68.78 $\pm$ 0.07
Linoleic ( $C_{18:2}$ )	15.91 $\pm$ 0.05	23.99 $\pm$ 0.06
Linolenic ( $C_{18:3}$ )	0.07 $\pm$ 0.02	0.07 $\pm$ 0.01
Arachidic ( $C_{20:0}$ )	0.07 $\pm$ 0.01	0.08 $\pm$ 0.03
Gadoleic ( $C_{20:1}$ )	0.05 $\pm$ 0.01	0.07 $\pm$ 0.02

Mean of two determinations

ND not detected

It is visible from the table that oleic acid ( $C_{18:1}$ ) was the most abundant fatty acid in both oils and accounted for 75.4 and 68.7% of the total fatty acids in PKO and AKO, respectively. Linoleic acid ( $C_{18:2}$ ) was the second main fatty acid in both oils and accounted for 15.9 and 23.9% of the total fatty acids in PKO and AKO, respectively. Regarding saturated fatty acids (SFA), palmitic acid ( $C_{16:0}$ ) was the main SFA in both oils and accounted for 5.83 and 5.12% of the total identified fatty acids in PKO and AKO, respectively. The fatty acid profile of PKO and AKO is found to be in similar with those reported in the literature [35]. These results are in good agreement with the fatty acid composition for several *Prunus* kernels described in the literature [26, 36]. Matthaeus and Ozcan [19] found the similar results for PKO and AKO, wherein  $C_{18:1}$ ,  $C_{18:2}$  and  $C_{16:0}$  levels were 63.9–78.5%; 9.7–26.9%, and 5.4–7.3% in PKO, and 62.3–71.6%; 18.7–28.0%, and 4.9–5.7% in AKO, respectively. Oleic (62.0–70.6%), linoleic (20.5–27.7%), palmitic (5.0–7.79%) and linolenic (0.4–1.42%) acids were present in major quantity of AKO [37]. In another study, the major fatty acids detected in AKO were oleic (62.3–80.9%), linoleic (13.1–30.3%) and palmitic (3.35–5.93%) acids [21]. Recently, palmitic, oleic and linoleic acids were determined as major fatty acids in AKO [14].

The fatty acid composition of PKA and AKO is highly favorable for human nutrition. High oleic-linoleic oils are of interest because of their stability and nutritional properties. Clinical trials reported that high oleic oils showed significant reductions in total cholesterol (TC), LDL-cholesterol, triacylglycerols, TC:HDL-cholesterol and increased HDL-cholesterol. High oleic oils have favorable impacts on plasma lipid risk factors and coronary heart disease risk [38]. Linoleic acid is considered as one of the most important polyunsaturated fatty acids (PUFA)

in human nutrition due to its preventive impacts for heart diseases [14, 38].

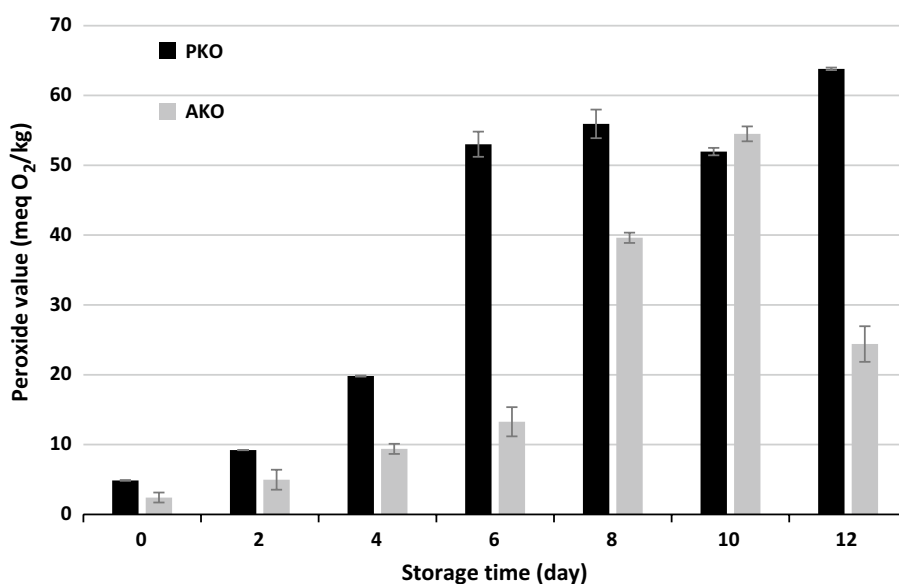
### Effect of thermal-oxidation on the stability of PKO and AKO

Changes in PV of PKO and AKO during the oven test at 60 °C were given in Fig. 1. The results indicated that PV tends to increase with the increasing of the storage time. At the end of 12th day of storage under the thermal oxidation condition, the PV of AKO and PKO were reached up to 24.41 and 63.82 meq O<sub>2</sub>/kg, respectively. The increment in PV for PKO was noted to be higher when compared to that of AKO. Individual headspace vials were used during

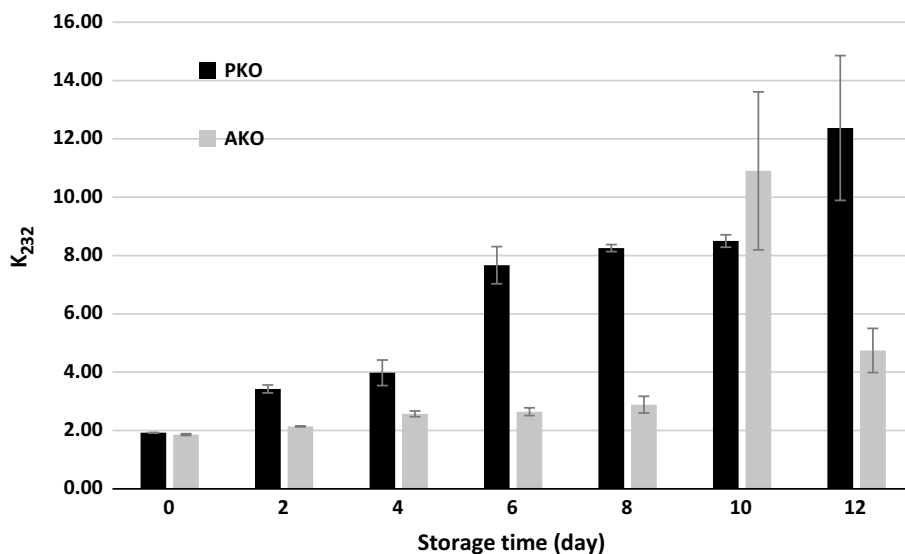
the experiment; hence the volatile compounds were kept in the vials. Durmaz et al. [29] studied the roasting effect on oxidative stability of AKO at 70 °C and showed that PV increased up to 80 meq O<sub>2</sub>/kg oil at the end of 22 days of storage. There were no significant differences in the levels of PV with the result reported for *n*-hexane-extracted PKO that stored up to 7 days at 60 °C [39].

K<sub>232</sub> values determined in the oil samples during thermal oxidation are shown in Fig. 2. After 8 days of storage under accelerated oxidation condition, K<sub>232</sub> values of PKO was increased rapidly in comparison with AKO. After 10 days, K<sub>232</sub> values of AKO exhibited an increase in comparison with PKO. However, at the end of storage period (12 days), K<sub>232</sub> value of PKO was increased over the K<sub>232</sub> value of

**Fig. 1** Changes in PV of oils during storage at thermal conditions. Error bars represent standard deviation obtained from duplicate experiments



**Fig. 2** Changes in K<sub>232</sub> values of oils during thermal oxidations. Error bars represent standard deviation obtained from duplicate experiments



**Table 2** Changes in the volatile compounds of PKO during storage under thermal conditions at 60 °C

Compound	KI	ID	Storage time (day)						
			0 (Fresh)	2	4	6	8	10	12
Octane	800	A	ND	ND	ND	7.47 ± 2.91c	43.88 ± 2.14b	70.82 ± 1.95a	24.58 ± 18.43bc
1-Pentanol	821	A	0.37 ± 0.06c	1.70 ± 0.04c	2.16 ± 0.04c	10.60 ± 1.97b	15.66 ± 0.33a	16.36 ± 1.82a	11.48 ± 1.85b
Hexanal	838	A	18.32 ± 0.30e	31.92 ± 0.63d	48.70 ± 6.77c	138.91 ± 11.36b	154.86 ± 2.11a	145.53 ± 0.29ab	149.24 ± 2.60ab
Ethylbenzene	886	A	1.01 ± 0.04abc	1.03 ± 0.00abc	1.09 ± 0.01ab	1.13 ± 0.03a	0.96 ± 0.01bc	0.79 ± 0.13d	0.94 ± 0.05c
<i>p</i> -xylene	894	A	3.30 ± 0.08b	3.44 ± 0.03b	3.61 ± 0.07b	4.36 ± 0.35a	4.46 ± 0.32a	4.44 ± 0.33a	4.26 ± 0.13a
Nonane	900	A	ND	ND	ND	0.49 ± 0.03d	0.97 ± 0.02b	1.32 ± 0.13a	0.71 ± 0.19c
Isoamyl acetate	905	C	1.18 ± 0.36b	1.00 ± 0.01b	1.84 ± 0.16a	ND	ND	ND	ND
2-Hexenal	906	A	ND	ND	ND	7.98 ± 1.42c	15.45 ± 0.26b	19.51 ± 1.27a	12.44 ± 3.60b
2-Heptanone	935	A	ND	ND	0.70 ± 0.12d	1.95 ± 0.44c	3.10 ± 0.13b	3.80 ± 0.18a	2.94 ± 0.28b
$\alpha$ -Thujene	939	A	8.38 ± 0.59bc	8.56 ± 0.16b	8.92 ± 0.15ab	9.77 ± 0.69a	8.64 ± 0.31b	7.38 ± 0.36c	8.50 ± 0.52b
heptanal	943	A	1.38 ± 0.22c	1.83 ± 0.03c	2.50 ± 0.25c	5.59 ± 1.17b	6.92 ± 0.10a	7.36 ± 0.50a	6.22 ± 0.08ab
$\alpha$ -Pinene	948	A	4.88 ± 0.08a	4.89 ± 0.11a	4.97 ± 0.05a	3.80 ± 0.23b	2.45 ± 0.09c	1.81 ± 0.18d	2.36 ± 0.50c
Pentanoic acid	970	C	0.23 ± 0.04c	0.41 ± 0.03c	0.62 ± 0.10c	3.92 ± 0.98b	5.42 ± 0.88a	5.45 ± 0.47a	6.23 ± 0.74a
$\beta$ -Pinene	996	A	0.85 ± 0.04	0.88 ± 0.02	0.91 ± 0.00	0.77 ± 0.00	0.77 ± 0.36	0.59 ± 0.05	0.60 ± 0.07
2,2,4,6,6-Penta-methylheptane	998	B	17.13 ± 0.78bc	17.81 ± 0.42b	18.45 ± 0.36ab	20.14 ± 1.58a	17.33 ± 1.29bc	15.04 ± 0.65c	16.97 ± 0.82bc
2-Pentylfuran	1011	B	0.57 ± 0.01d	0.80 ± 0.04d	1.12 ± 0.10d	4.79 ± 2.24cd	9.61 ± 0.21b	14.50 ± 2.50a	7.85 ± 3.56bc
<i>E</i> -2-heptenal	1012	A	0.24 ± 0.04d	0.81 ± 0.03d	2.38 ± 0.75d	26.25 ± 5.35c	44.76 ± 3.94ab	54.91 ± 4.60a	41.83 ± 9.55b
Benzaldehyde	1023	A	48.82 ± 4.21c	53.41 ± 1.94bc	56.77 ± 1.15bcd	74.32 ± 11.68a	67.29 ± 6.18abc	60.00 ± 6.42abcd	71.30 ± 1.68ab
1-Octen-3-ol	1027	B	ND	ND	ND	4.89 ± 1.58b	7.41 ± 1.01a	8.86 ± 0.77a	7.55 ± 1.04a
2-Octanone	1037	A	ND	ND	ND	0.62 ± 0.30b	0.87 ± 0.03ab	1.18 ± 0.22a	0.95 ± 0.05ab
Octanal	1046	B	2.90 ± 0.26b	3.25 ± 0.12b	3.32 ± 0.06b	5.19 ± 1.31a	5.88 ± 0.38a	6.23 ± 0.41a	6.17 ± 0.88a
<i>p</i> -cymene	1052	A	8.54 ± 0.99d	9.31 ± 0.38d	9.90 ± 0.25cd	13.84 ± 2.00a	12.22 ± 1.60abc	10.90 ± 1.02bcd	13.24 ± 0.25ab
Hexanoic acid	1062	B	1.45 ± 0.35b	2.95 ± 0.18b	3.39 ± 0.04b	9.60 ± 2.28a	12.17 ± 2.02a	12.49 ± 0.83a	12.99 ± 2.33a
2,4-Heptadienal	1076	B	ND	ND	ND	0.78 ± 0.16c	1.07 ± 0.06ab	1.23 ± 0.08a	0.98 ± 0.17bc
3-Octen-2-one	1094	B	ND	ND	ND	1.32 ± 0.35b	2.00 ± 0.27a	2.08 ± 0.17a	2.04 ± 0.44a
2-Octenal	1118	B	ND	ND	ND	2.37 ± 0.82b	5.63 ± 1.03a	7.56 ± 0.63a	6.03 ± 2.06a
Benzyl alcohol	1121	A	8.03 ± 1.70d	11.90 ± 0.57bcd	12.05 ± 0.03bcd	17.91 ± 3.06a	14.68 ± 3.44abc	10.75 ± 0.67cd	16.38 ± 0.63ab
Nonanal	1153	A	1.02 ± 0.19b	1.22 ± 0.06b	1.43 ± 0.10b	4.78 ± 1.25a	6.24 ± 1.50a	6.02 ± 0.47a	6.68 ± 0.01a
octanoic acid	1253	B	ND	0.39 ± 0.01c	0.43 ± 0.01c	0.92 ± 0.12b	1.12 ± 0.19ab	0.97 ± 0.10ab	1.25 ± 0.20a
2,4-Nonadienal <sup>a</sup>	1268	B	ND	ND	ND	0.24 ± 0.04b	0.25 ± 0.06b	0.22 ± 0.02b	0.38 ± 0.03a
2,4-Nonadienal	1288	B	ND	ND	ND	0.50 ± 0.10b	0.59 ± 0.17ab	0.51 ± 0.06b	0.78 ± 0.11a
2-Decenal	1327	B	ND	ND	ND	0.50 ± 0.15b	0.85 ± 0.23a	1.02 ± 0.11a	0.90 ± 0.23a
Nonanoic acid	1347	B	ND	ND	ND	0.32 ± 0.02b	0.48 ± 0.11a	0.47 ± 0.07a	0.56 ± 0.05a
<i>E,Z</i> -2,4-decadienal	1368	B	ND	ND	ND	ND	0.32 ± 0.09b	0.51 ± 0.06a	0.33 ± 0.10b
<i>E,E</i> -2,4-decadienal	1393	B	ND	ND	ND	ND	0.41 ± 0.12ab	0.57 ± 0.07a	0.27 ± 0.23b

Results are expressed as means ± standard deviations of total ion current (TIC) area units ( $\times 10^{-6}$ ); values are means ± standard deviations of two measurements ( $n = 2$ ) with different letters in the same row indicating significant differences at  $p < 0.05$

KI Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4  $\mu$ m film thickness) installed on a gas chromatograph equipped with a mass-selective detector, RI reliability of identification, A mass spectrum and retention time identical with authentic sample, B mass spectrum and Kovats index from literature in accordance, C tentative identification by mass spectrum, ND not detected

<sup>a</sup> Isomers of this compound was not identified

AKO. The similar changes in the  $K_{232}$  values were recorded in soybean oil during storage under thermal condition [40]. This may be attributed to the fact that the fluctuations in the  $K_{232}$  value stem from the conjugated dienes appeared at the beginning stage of oxidation so that the dienes prone to degrade into smaller ones due to the unstable nature.

A total of 19 compounds were identified in the fresh PKO before thermal oxidation (Table 2). The identified compounds in PKO consisted of 6 aldehydes (hexanal, heptanal, *E*-2-heptenal, benzaldehyde, octanal, and nonanal), 4 terpenes ( $\alpha$ -thujene,  $\alpha$ -pinene,  $\beta$ -pinene, and *p*-cymene), 3 hydrocarbons (ethylbenzene, *p*-xylene, and

2,2,4,6,6-pentamethylheptane), 2 alcohols (1-pentanol, and benzyl alcohol), 2 acids (pentanoic acid, and hexanoic acid), 1 ester (isoamyl acetate), 1 furan (2-pentylfuran). Benzaldehyde with the range of  $48.82 \times 10^6$  AU was the major compound of volatiles in the fresh PKO. Hexanal was the second major compound accounted for  $18.32 \times 10^6$  AU in PKO. The major compounds detected in the fresh PKO were in agreement with results reported by Nunes et al. [41] and Pino et al. [42]. It is to be emphasized that the branched hydrocarbon (2,2,4,6,6-pentamethylheptane) previously found among the volatiles of Iberian pig lipids [43] is identified for the first time in PKO.

Throughout the thermal oxidation of PKO, the volatile compounds including 2 hydrocarbons (octane and nonane), 3 ketones (2-heptanone, 2-octanone, and 3-octen-2-one), 8 aldehydes (2-hexenal, 2,4-heptadienal, 2-octenal, 2,4-nonadienal isomers, 2-decenal, *E,Z*-2,4-decadienal, and *E,E*-2,4-decadienal) and 2 acids (octanoic, and nonanoic) were released constantly. Most of these compounds previously mentioned were appeared after 6 days of storage under thermal oxidation conditions. Of those compounds, 2-heptanone was first appeared as a volatile oxidation compound. *E,Z*-2,4-decadienal, and *E,E*-2,4-decadienal became apparent towards the end of storage. 2,4-decadienal was a decomposition product from heat-oxidized linoleate [44]. *E,Z*-2,4-decadienal, and *E,E*-2,4-decadienal were detected in high values upon frying of *Pinus halepensis* seed oil [45]. Among the compounds resulted from the thermal oxidation of PKO during storage at 60 °C, octane being released after 6 days of storage, wherein the level of octane reached the value of 70.82 AU at the 10th day of storage. Hexanal is resulted from the auto-oxidation of linoleic acid [46]. According to the experimental findings, a significant ( $p < 0.05$ ) increase in the total amount of hexanal was observed during the thermal oxidation. After 6 days, there was a sharp increase in hexanal level from the initial average value of  $18.32 \times 10^6$  area units to  $154.86 \times 10^6$  area units in PKO.

*E*-2-heptenal is another major volatile compound stemming from the oxidation process of PKO due to the decomposition product of linoleate hydroperoxides [47]. In the current work, the level of *E*-2-heptenal compound was found to augment sharply with the increase in the thermal condition period for PKO (especially between the 4th day and the 6th day of storage). Our experimental results agree with the results of Gómez-Alonso et al. [48] who reported that hexanal and *E*-2-heptenal were determined as the major volatile oxidation compounds in the purified olive oil with high oleic acid content.

The volatile compounds measured in AKO during thermal oxidation are shown in Table 3. A total of nine volatile compounds were identified in the AKO before treatment.

The major volatile compound found in the headspace of AKO was benzaldehyde, and hexanal was the second most common volatile compound. Benzaldehyde, exhibiting a very strong almond aroma, was found in a level of  $58.40 \times 10^6$  AU in the AKO [49]. As for the hexanal contribution to the apricot aroma, the amount of volatile compound was observed to be  $47.80 \times 10^6$  AU. The major volatile compounds of AKO found in this study were also reported in the apricot oil [24] and the volatile compounds have already been assigned in the apricots [50]. The content of volatile oxidation compounds was found to be in the highest level in the oxidized AKO after 10 days of storage. After 10 days of storage, hexanal with the value of  $199.28 \times 10^6$  AU was the dominant volatile component in the AKO. *E*-2-heptenal with the value of  $58.88 \times 10^6$  AU was found to be the second most volatile component in AKO. Further, 2-heptanone, heptanal, pentanoic acid, 2-pentylfuran, octanal, hexanoic acid, 3-octen-2-one and nonanal were detected, and those volatile compounds release increased up to their high values after the 10 days of storage. On the other hand, the volatile oxidation compounds of 1-pentanol, 2-hexenal, pentanoic acid, 1-octen-3-ol, 2-octanone, 2,4-heptadienal, 2-octenal, octanoic acid, 2,4-nonadienal, 2-decenal, and 2,4-decadienal were only detected after 10 days of storage under thermal conditions. The volatiles of hexanal and octane accounted for  $4.1 \times 10^3$  AU in the olive oil after 8 days of storage at 60 °C [51].

#### Effect of photo-oxidation on the stability of PKO and AKO

Changes in the levels of PV of PKO and AKO upon photo-oxidation is shown in Fig. 3. It is clear from the figure that PV of oil samples were sharply increased. At the end of 12 days of storage, PV of AKO and PKO reached up to 67.62 and 117.5 meq O<sub>2</sub>/kg, respectively. During the storage time, the increase in PV value of PKO was higher than that of AKO. It could be noted that the impact of light irradiation on the increment of PV of AKO and PKO is higher than that of the thermal oxidation. Jung et al. [52] reported that the increasing rate of PV for soybean oil stored under light irradiation conditions was slightly greater than that of soybean oil stored under thermal conditions (60 °C). This difference is related to the photosensitizers, namely the chlorophylls, that act as photosensitizer in photosynthesis of carbohydrates in plants and transfer the energy absorbed from light into the triplet oxygen that resulted in the formation of reactive radicals such as singlet oxygen [53]. Bianchi et al. [54] determined the influences of different light wavelengths with varied irradiation powers on the chlorophyll of soybean oil and reported a rapid increment in PV values of soybean oil with the decrement in the wavelength.

**Table 3** Changes in the volatile compounds of AKO during storage under thermal conditions at 60 °C

Compound	KI	ID	Storage time (day)							
			0 (Fresh)	2	4	6	8	10	12	
Octane	800	A	ND	ND	ND	ND	ND	ND	35.52 ± 6.75	ND
1-Pentanol	821	A	ND	ND	ND	ND	ND	ND	16.46 ± 7.59	ND
Hexanal	838	A	47.80 ± 0.99c	58.78 ± 0.24c	62.30 ± 1.63bc	64.41 ± 4.93bc	59.79 ± 0.07c	199.28 ± 8.53a	80.33 ± 18.22b	
Furfural	896	C	2.24 ± 0.02ab	2.28 ± 0.06ab	2.28 ± 0.13ab	2.27 ± 0.15ab	2.32 ± 0.21ab	2.40 ± 0.26a	1.98 ± 0.02b	
Isoamyl acetate	905	C	1.19 ± 0.03c	1.88 ± 0.06b	1.98 ± 0.08b	2.06 ± 0.08ab	2.07 ± 0.19ab	ND	2.23 ± 0.02a	
2-Hexenal	906	A	ND	ND	ND	ND	ND	ND	14.68 ± 5.88	ND
2-Heptanone	935	A	0.69 ± 0.01b	0.73 ± 0.06b	0.91 ± 0.09b	0.87 ± 0.13b	1.05 ± 0.26b	10.07 ± 4.37a	1.33 ± 0.38b	
heptanal	943	A	0.78 ± 0.00b	1.09 ± 0.10b	1.29 ± 0.04b	1.26 ± 0.17b	1.38 ± 0.10b	4.40 ± 0.78a	1.31 ± 0.04b	
Pentanoic acid	970	B	ND	ND	ND	ND	ND	ND	2.65 ± 0.94	ND
2-Pentylfuran	1011	B	0.68 ± 0.03	1.03 ± 0.07	1.53 ± 0.04	1.07 ± 0.21	1.25 ± 0.07	14.77 ± 2.96	1.83 ± 0.35	
<i>E</i> -2-heptenal	1012	A	ND	ND	ND	0.46 ± 0.22b	0.36 ± 0.07b	58.88 ± 28.40a	2.63 ± 1.54b	
Benzaldehyde	1023	A	58.40 ± 2.38a	53.70 ± 1.79b	53.84 ± 6.75b	50.83 ± 4.07b	61.21 ± 6.14ab	66.41 ± 0.76a	53.07 ± 3.99b	
1-Octen-3-ol	1027	B	ND	ND	ND	ND	ND	ND	10.46 ± 5.76	ND
2-Octanone	1037	A	ND	ND	ND	ND	ND	ND	1.16 ± 0.48	ND
Octanal	1046	B	ND	0.78 ± 0.04b	0.72 ± 0.10b	0.67 ± 0.07b	0.88 ± 0.09b	2.55 ± 0.60a	0.76 ± 0.09b	
<i>p</i> -cymene	1052	A	ND	0.46 ± 0.07b	0.39 ± 0.06b	0.31 ± 0.14b	0.48 ± 0.05b	0.77 ± 0.03a	0.46 ± 0.08b	
Hexanoic acid	1062	C	1.29 ± 0.16b	2.11 ± 0.10b	1.74 ± 0.42b	1.48 ± 0.32b	2.05 ± 0.34b	21.05 ± 9.37a	2.10 ± 0.09b	
2,4-Heptadienal	1076	B	ND	ND	ND	ND	ND	ND	1.57 ± 1.00	ND
3-Octen-2-one	1094	B	ND	ND	0.22 ± 0.03b	0.17 ± 0.05b	0.26 ± 0.05b	2.65 ± 0.93a	0.39 ± 0.06b	
2-Octenal	1118	B	ND	ND	ND	ND	ND	ND	9.99 ± 5.46	ND
Benzyl alcohol	1121	A	ND	3.32 ± 0.20a	2.44 ± 0.52ab	2.00 ± 0.32b	3.03 ± 0.44ab	2.86 ± 0.73ab	1.90 ± 0.66b	
Nonanal	1153	A	0.41 ± 0.03e	0.61 ± 0.03cd	0.57 ± 0.07de	0.52 ± 0.10de	0.75 ± 0.11bc	5.42 ± 0.06a	0.91 ± 0.06b	
2-Nonenal	1224	B	ND	ND	ND	ND	ND	ND	0.41 ± 0.15	ND
2,4-Nonadienal	1288	B	ND	ND	ND	ND	ND	ND	0.22 ± 0.04	ND
2-Decenal	1327	B	ND	ND	ND	ND	ND	ND	0.54 ± 0.30	ND
<i>E,E</i> -2,4-decadienal	1393	B	ND	ND	ND	ND	ND	ND	0.45 ± 0.04	ND

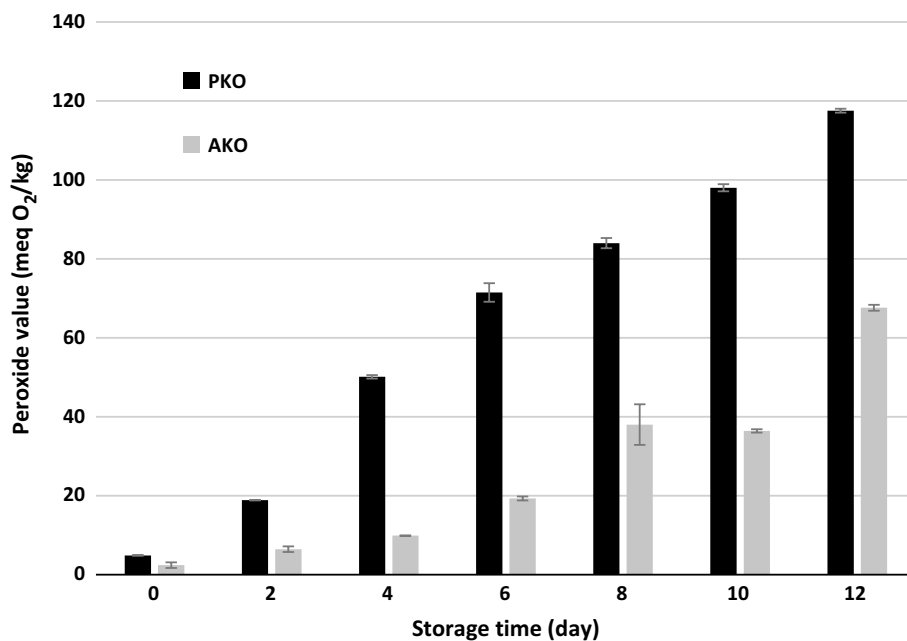
Results are expressed as means ± standard deviations of total ion current (TIC) area units ( $\times 10^{-6}$ ); values are means ± standard deviations of two measurements ( $n = 2$ ) with different letters in the same row indicating significant differences at  $p < 0.05$

*KI* Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4  $\mu\text{m}$  film thickness) installed on a gas chromatograph equipped with a mass-selective detector, *RI* reliability of identification, *A* mass spectrum and retention time identical with authentic sample, *B* mass spectrum and Kovats index from literature in accordance, *C* tentative identification by mass spectrum, *ND* not detected

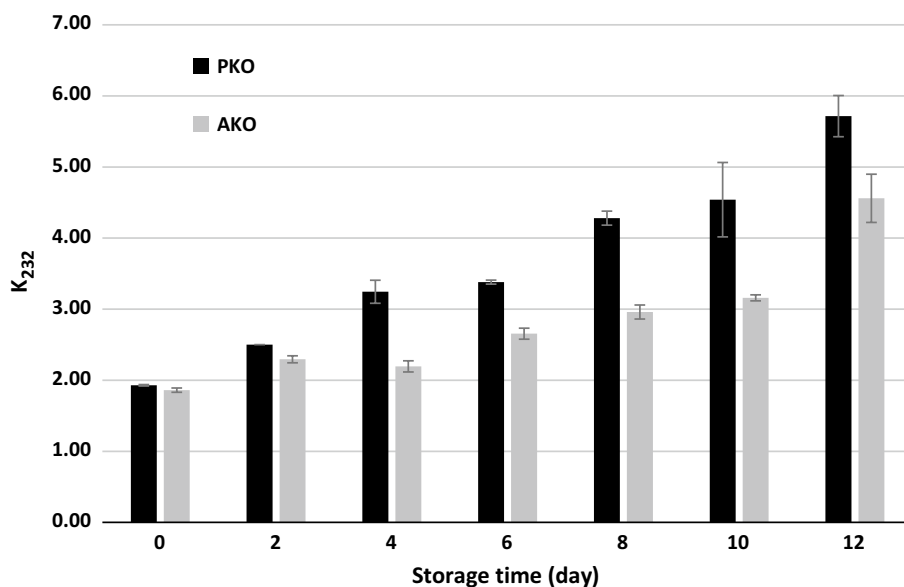
The  $K_{232}$  values for both AKO and PKO exhibited a linear increase during photo-oxidation (Fig. 4). In this respect,  $K_{232}$  values in PKO and AKO increased from 1.93 and 1.86 (fresh oils) to 5.72 and 4.75 after 12 days of storage, respectively. It is noteworthy that the increase rate in  $K_{232}$  of PKO was higher as compared to AKO during the storage in the existence of light irradiation. The similar increase was observed for PV belonging to both oils. The increment in conjugated diene values was found to be higher in the soybean oil exposed to the sunlight than that of soybean oil stored at the dark [55].

Among the volatile oxidation products in PKO, hexanal and *E*-2-heptenal amounts were increased with the prolonged storage time under photo-oxidation conditions (Table 4). The level of hexanal reached  $48.95 \times 10^6$  AU, while the level of *E*-2-heptenal increased up to  $20.13 \times 10^6$  AU at the end of storage. *E*-2-heptenal is an important volatile aldehyde induced during linoleic acid oxidation wherein the odor threshold of this compound is very low with 0.001 mg/kg [31]. The other volatile components including 1-octene, octane and 1-octen-3-ol were detected after 10 days of storage. The release of nonanoic

**Fig. 3** Changes in PV of oils during storage at photo-oxidation conditions. *Error bars* represent standard deviation obtained from duplicate experiments



**Fig. 4** Changes in  $K_{232}$  values of oils during storage at photo-oxidation conditions. *Error bars* represent standard deviation obtained from duplicate experiments



acid was detected only at the end of storage period. Lee and Min [56] reported that *E*-2-heptenal, 1-octen-3-ol, and octanoic acid were measured in a linoleic acid model stored under light irradiation. Other research group reported that *E*-2-heptenal was detected to be more abundant in olive oil exposed to light irradiation [57].

Hexanal, 1-octene, and *E*-2-heptenal were the major volatile oxidation compounds detected in AKO stored under the light irradiation conditions (Table 5). Changes in hexanal, and *E*-2-heptenal levels were noticed to be

close to each other for both oils until the tenth day of storage. After 12 days of storage, the amounts of hexanal, and *E*-2-heptenal sharply increased up to  $100.01 \times 10^6$  and  $11.57 \times 10^6$  AU, respectively. Interestingly, 1-octene ( $11.94 \times 10^6$  AU) was detected only at 12th day of storage. Similarly, 1-octen-3-ol, 2,4-heptadienal, and 2-octenal were determined at the end of storage. On the other hand, 2-heptanone, heptanal, 2-octanone, octanal, hexanoic acid, and nonanal levels were observed to be increased regularly during the experimental process.



**Table 4** Changes in the volatile compounds of PKO during storage under photooxidation conditions

Compound	KI	ID	Storage time (day)						
			0 (Fresh)	2	4	6	8	10	12
1-Octene	790	A	ND	ND	ND	ND	ND	9.15 ± 0.20	11.90 ± 0.02
Octane	800	A	ND	ND	ND	ND	ND	7.81 ± 0.46	14.98 ± 0.14
Hexanal	838	A	18.32 ± 0.30c	18.06 ± 0.35c	17.01 ± 0.14c	19.06 ± 0.11c	20.25 ± 0.18c	25.99 ± 0.87b	48.95 ± 4.21a
Ethylbenzene	886	A	1.01 ± 0.04c	1.03 ± 0.00c	1.13 ± 0.00b	1.26 ± 0.02a	1.24 ± 0.00a	1.30 ± 0.10a	1.27 ± 0.04a
<i>p</i> -xylene	894	A	3.30 ± 0.08d	3.38 ± 0.03d	3.70 ± 0.03c	4.44 ± 0.08ab	4.31 ± 0.01b	4.70 ± 0.31a	4.55 ± 0.16ab
Isoamyl acetate	905	C	1.18 ± 0.36d	0.95 ± 0.02d	1.80 ± 0.01c	2.08 ± 0.02bc	2.09 ± 0.00bc	2.28 ± 0.17b	2.95 ± 0.05a
1-Hexanol	921	A	ND	5.79 ± 0.17b	6.55 ± 0.13ab	7.68 ± 0.18a	6.33 ± 0.06b	6.99 ± 1.17ab	6.70 ± 0.44ab
2-Heptanone	935	A	ND	ND	0.58 ± 0.01d	1.14 ± 0.01bc	0.76 ± 0.01cd	1.23 ± 0.45b	1.65 ± 0.03a
$\alpha$ -Thujene	939	A	8.38 ± 0.59ab	8.08 ± 0.18ab	8.13 ± 0.06ab	9.01 ± 0.25a	7.95 ± 0.03b	8.74 ± 0.81ab	9.03 ± 0.28a
Heptanal	943	A	1.38 ± 0.22d	1.32 ± 0.03d	1.32 ± 0.01d	1.76 ± 0.03c	1.56 ± 0.00cd	2.10 ± 0.17b	3.26 ± 0.02a
$\alpha$ -Pinene	948	A	4.88 ± 0.08b	4.89 ± 0.05b	5.02 ± 0.03b	5.29 ± 0.08a	4.90 ± 0.03b	4.91 ± 0.08b	4.19 ± 0.00c
Pentanoic acid	970	B	0.23 ± 0.04d	0.26 ± 0.01d	0.45 ± 0.02c	0.62 ± 0.04b	0.55 ± 0.04bc	0.64 ± 0.09b	0.80 ± 0.05a
$\beta$ -Pinene	996	A	0.85 ± 0.04b	0.89 ± 0.01b	0.94 ± 0.01b	1.06 ± 0.05a	0.92 ± 0.01b	1.03 ± 0.04a	0.85 ± 0.06b
2,2,4,6,6-Pentamethylheptane	998	B	17.13 ± 0.78c	17.35 ± 0.12c	18.98 ± 0.20bc	22.46 ± 0.49a	20.09 ± 0.12b	22.08 ± 1.36a	20.61 ± 1.17ab
2-Pentylfuran	1011	B	0.57 ± 0.01	ND	ND	ND	ND	ND	ND
<i>E</i> -2-heptenal	1012	A	0.24 ± 0.04f	0.39 ± 0.01f	1.17 ± 0.11e	3.70 ± 0.28d	6.10 ± 0.30c	11.98 ± 0.15b	20.13 ± 0.48a
Benzaldehyde	1023	A	48.82 ± 4.21ab	46.68 ± 0.14b	49.45 ± 0.04ab	54.83 ± 3.08a	44.04 ± 0.80b	41.91 ± 6.01b	31.68 ± 2.68c
1-Octen-3-ol	1027	B	ND	ND	ND	ND	ND	2.69 ± 0.04	4.52 ± 0.20
2-Octanone	1037	A	ND	ND	0.44 ± 0.00d	0.60 ± 0.00c	0.58 ± 0.06c	0.89 ± 0.06b	1.07 ± 0.06a
octanal	1046	A	2.90 ± 0.26cd	2.71 ± 0.04d	2.94 ± 0.07cd	3.76 ± 0.14b	3.19 ± 0.08c	3.86 ± 0.16b	5.02 ± 0.06a
<i>p</i> -cymene	1052	A	8.54 ± 0.99c	8.43 ± 0.16c	10.40 ± 0.07bc	14.00 ± 0.46a	11.96 ± 0.06ab	14.04 ± 1.47a	13.85 ± 1.38a
Hexanoic acid	1062	B	1.45 ± 0.35f	2.44 ± 0.22e	3.55 ± 0.00d	4.84 ± 0.03b	4.27 ± 0.12c	4.94 ± 0.04b	7.22 ± 0.12a
Benzyl alcohol	1121	A	8.03 ± 1.70d	9.98 ± 0.71d	13.78 ± 0.17c	19.22 ± 0.36a	15.30 ± 0.03bc	17.86 ± 2.25ab	20.91 ± 3.03a
Nonanal	1153	A	1.02 ± 0.19d	0.88 ± 0.07d	1.04 ± 0.00d	1.40 ± 0.03c	1.36 ± 0.09c	1.66 ± 0.06b	3.05 ± 0.04a
Octanoic acid	1253	B	ND	0.28 ± 0.02d	0.35 ± 0.00c	0.45 ± 0.00b	0.46 ± 0.02b	0.47 ± 0.00b	0.77 ± 0.03a
2-Decenal	1327	B	ND	ND	ND	ND	0.19 ± 0.02c	0.26 ± 0.03b	0.61 ± 0.05a
Nonanoic acid	1347	B	ND	ND	ND	ND	ND	ND	0.40 ± 0.04

Results are expressed as means  $\pm$  standard deviations of total ion current (TIC) area units ( $\times 10^{-6}$ ); values are means  $\pm$  standard deviations of two measurements ( $n = 2$ ) with different letters in the same row indicating significant differences at  $p < 0.05$

*KI* Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4  $\mu$ m film thickness) installed on a gas chromatograph equipped with a mass-selective detector, *RI* reliability of identification, *A* mass spectrum and retention time identical with authentic sample, *B* mass spectrum and Kovats index from literature in accordance, *C* tentative identification by mass spectrum, *ND* not detected

## Conclusion

The results of the present study provide important information on the volatile oxidation products produced in cold-pressed AKO and PKO during thermal and photo-oxidation. The increments in  $K_{232}$  and PV values of PKO were higher than those in AKO. In fact, light irradiation conditions resulted in more PV and  $K_{232}$  values than the thermal oxidation did. PV values of PKO reached the maximum value after 12 days of storage, while PV

values of AKO reached the maximum value after 10 days of storage under thermal oxidation condition.  $K_{232}$  values exhibited the similar behavior under accelerated thermal oxidation condition. Under the photo-oxidation conditions, both PV and  $K_{232}$  values recorded similar behavior. There were no literature reports on the effects of thermal and photo-oxidation conditions on the volatile oxidation compounds of cold-pressed AKO and PKO, therefore, no comparison could be done with the literatures. The current study pointed out that hexanal and *E*-2-heptenal

**Table 5** Changes in the volatile compounds of AKO during storage under photooxidation conditions

Compound	KI	ID	Storage time (day)						
			0 (Fresh)	2	4	6	8	10	12
1-octene	790	A	ND	ND	ND	ND	ND	ND	11.94 ± 0.69
Hexanal	838	A	47.80 ± 0.99b	48.87 ± 0.42b	50.43 ± 0.03b	48.46 ± 0.22b	49.46 ± 0.42b	48.79 ± 0.71b	100.01 ± 20.63a
Furfural	896	B	2.24 ± 0.02ab	2.30 ± 0.04a	2.36 ± 0.04a	2.06 ± 0.07bc	2.01 ± 0.08c	1.92 ± 0.04c	1.73 ± 0.16d
Isoamyl acetate	905	C	1.19 ± 0.03c	2.07 ± 0.11b	2.23 ± 0.01b	2.10 ± 0.09b	2.20 ± 0.08b	2.26 ± 0.11b	2.54 ± 0.15a
2-Heptanone	935	A	0.69 ± 0.01b	0.69 ± 0.02b	0.75 ± 0.03b	0.75 ± 0.00b	0.81 ± 0.02b	0.85 ± 0.02b	1.47 ± 0.66a
Heptanal	943	A	0.78 ± 0.00b	0.95 ± 0.18b	0.89 ± 0.03b	0.84 ± 0.01b	0.94 ± 0.00b	1.00 ± 0.04b	1.92 ± 0.32a
2-Pentylfuran	1011	B	0.68 ± 0.03	0.48 ± 0.03	ND	ND	ND	ND	ND
<i>E</i> -2-heptenal	1012	A	ND	ND	0.51 ± 0.08b	0.94 ± 0.12b	1.62 ± 0.30b	2.36 ± 0.08b	11.57 ± 2.16a
Benzaldehyde	1023	A	58.40 ± 2.38a	52.94 ± 4.38a	55.18 ± 3.28a	43.41 ± 2.46b	43.07 ± 3.54b	43.27 ± 4.13b	27.06 ± 4.56c
1-Octen-3-ol	1027	B	ND	ND	ND	ND	ND	ND	2.22 ± 0.42
2-Octanone	1037	A	ND	ND	ND	ND	ND	0.34 ± 0.00	0.77 ± 0.12
Octanal	1046	B	ND	0.70 ± 0.07b	0.77 ± 0.05b	0.73 ± 0.00b	0.83 ± 0.02b	0.87 ± 0.05b	2.24 ± 0.51a
Hexanoic acid	1062	B	1.29 ± 0.16b	1.69 ± 0.18b	1.78 ± 0.09b	1.97 ± 0.09b	2.17 ± 0.05b	2.37 ± 0.22b	6.24 ± 1.33a
2,4-Heptadienal	1076	B	ND	ND	ND	ND	ND	ND	0.54 ± 0.09
2-Octenal	1118	B	ND	ND	ND	ND	ND	ND	1.77 ± 0.80
Benzyl alcohol	1121	A	ND	3.01 ± 0.49b	3.18 ± 0.16b	3.04 ± 0.15b	3.55 ± 0.14b	2.98 ± 0.06b	4.28 ± 0.22a
Nonanal	1153	A	0.41 ± 0.03b	0.44 ± 0.05b	0.47 ± 0.02b	0.54 ± 0.01b	0.68 ± 0.01b	0.73 ± 0.05b	2.44 ± 0.54a
Phenylethyl alcohol	1210	A	ND	0.51 ± 0.04c	0.62 ± 0.09c	0.52 ± 0.01c	1.07 ± 0.13b	1.02 ± 0.02b	1.48 ± 0.03a

Results are expressed as means ± standard deviations of total ion current (TIC) area units ( $\times 10^{-6}$ ); values are means ± standard deviations of two measurements ( $n = 2$ ) with different letters in the same row indicating significant differences at  $p < 0.05$

KI Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4  $\mu$ m film thickness) installed on a gas chromatograph equipped with a mass-selective detector, RI reliability of identification, A mass spectrum and retention time identical with authentic sample, B mass spectrum and Kovats index from literature in accordance, C tentative identification by mass spectrum, ND not detected

released during thermal-oxidation and photo-oxidation could be considered as good indicators for PKO and AKO stability.

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#### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

**Compliance with ethics requirements** This article does not contain any studies with human or animal subjects.

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