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Storage stability of whole-split pistachio nuts (*Pistachia vera* L.) at various conditions

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Abstract

Oil characteristics and chemical composition of whole-split pistachio nuts (*Pistachia vera* L.) were determined. Storage stability of the nuts was examined on the samples stored in modified atmosphere (2% air, 98% CO_2), air at the monolayer value at 10, 20, 30°C, and at ambient conditions. Most oxidation was observed under ambient storage conditions. CO_2 improved the storage stability especially at low temperatures. From the reaction rate constants of peroxide formation, it was revealed that as temperature increased, the ratio of rate constants (air/CO_2) approached to 1 which means that no significant difference existed between air and CO_2 storages at 30°C. The oxidation activation energies were 8.33 and 13.39 kcal/mol under air and CO_2 storage, respectively.

1. Introduction

The pistachio nut (*Pistachia vera* L.) is one of the favourite tree nuts of the world and is widely cultivated in saline, dry and hot areas of the Middle East, Mediterranean countries and USA. It contributes substantially to the agricultural exports of some of these countries (Garcia, Agar, & Streif, 1992; Shokraii, 1977). Several species of the genus *Pistacia* are referred to as pistachio, but only the fruits of *Pistacia vera* attain sufficiently large size to be acceptable to consumers as edible nuts (Shokraii & Esen, 1988).

The kernels are a rich source of oil (50–60%) and contain linolenic and linoleic fatty acids, essential for human diet and oleic acid (Garcia et al., 1992; Maskan & Karataş, 1998). They are highly nutritious and are eaten raw or fried with pepper and salt, and form a popular dessert in the Orient and Europe. They are used as a flavoring in cookery and confectionery and also favoured, because of the deep green colour of their kernels, in the ice cream and pastry industries. The pistachio nut tree bears a heavy crop of nuts one year and little or none the next (Woodroof, 1967).

Deterioration of fat-containing foods by oxidation is a major concern during storage of dehydrated foods and constitutes one of the most important technical problems of the food industries. Oxidative rancidity is the most important type of fat spoilage because all edible fats, as such, or as components of foods, contain unsaturated triglycerides. Oxidative deterioration of fat results in the development of a pungent and offensive off-flavour and the destruction of vitamins (A, D, E, K and C), essential fatty acids, chlorophylls, carotenes, amino acids, proteins, or enzymes by the production of toxic or physiologically active compounds (Angelo & Ory, 1975; Gardner, 1979) and is believed to lead to deteriorative processes in man, including aging (Karel, 1992; Pearson, Gray, Wolzak, & Horenstein, 1983).

One of the factors that affects the autoxidation of common food fats is the total number of unsaturated linkages in the sample. However, the total amount of unsaturation may not be as important as the degree of unsaturation within a given molecule. A fat high in linoleic or linolenic acid would be more susceptible to oxidation than one containing a similar amount of oleic acid. Oxygen is necessary for autoxidation of fats. At very low oxygen pressures, the rate of oxidation is slow. Therefore, the removal of atmospheric oxygen from a fat or food product exerts a protective effect (Aurand & Woods, 1973; Swern, 1964).

A common method of controlling the oxidation reaction is to reduce O_2 concentration in the storage atmosphere over the food by vacuum or nitrogen filling for dry or intermediate moisture foods (Kacyn, Saguay, &

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Karel, 1983) or CO_2 filling, as a biostat, for fresh fruits and vegetables, meat, chicken, fish and bakery products to prevent anaerobic microbial growth and lipid oxidation (Hotchkiss, 1988; Lioutas, 1988). The concentrations of gases (O_2, CO_2 or N_2) should be tailored to each individual product. Kacyn et al. (1983) reported that, for a model consisting of Avicel microcrystalline cellulose and methyl linoleate, O_2 concentration of below 2% affects rate constant dramatically with the rate decreasing sharply as the O_2 is decreased. Waletzko and Labuza (1976) used 0.5% O_2 in their study for an intermediate moisture food. However, Brecht (1980) proposed 0.0% O_2 and 100% CO_2 for tree nuts. It should also be pointed out that exposure to light and other radiations will catalyze initiations and should therefore be avoided. Other initiators, often neglected, are the volatile decomposition products of oxidation. Therefore, even the presence in the same container of an already oxidized food will tend to speed up the oxidation of an unoxidized food (Karel, 1992).

Another method of controlling lipid oxidation is storing food products at their monolayer moisture content (M_o) providing optimal stability of dehydrated foods (Hill & Rizvi, 1982; Labuza, Tannenbaum, & Karel, 1970). The effects are related to the fact that the water present at the M_o is tightly bound and cannot act as an aqueous-phase reaction medium and/or that the rate of reaction is so slow in this water as to be negligible in terms of food storage stability (Rockland, 1969; Labuza, 1980).

The objectives of this investigation were: (1) to determine the chemical composition and oil characteristics, (2) to determine storage stability of whole-split pistachio nuts (*Pistachia vera* L.) stored at various storage conditions and (3) to apply the Arrhenius model in prediction of peroxide formation at three constant temperatures and ambient conditions before using kinetic analysis to predict the rate and extent of reaction during storage.

2. Materials and methods

2.1. Reagents

All chemicals used in this study were supplied from Merck and Sigma Chemical Companies.

2.2. Pistachio nut samples

The Gaziantep variety of pistachio nuts was used for the experiments. The whole-split pistachio nut samples were received with an average moisture content of about 14% (wet basis) initially; samples were dried to 4% (wet basis) moisture content in a vacuum oven at 50°C then immediately stored in air-tight sealed jars under different conditions.

2.3. Storage

Storage was under ambient conditions (20–30°C, 27–92% relative humidity, RH), and at monolayer moisture content determined by Maskan and Karataş (1997) under air and 98% CO_2 gases at 10, 20, and 30°C. The details of preparation of modified storage atmosphere were reported by Maskan and Karataş (1998).

2.4. Chemical analysis

Moisture, oil, free fatty acid and protein (N×5.30) contents, and iodine value were determined according to the methods of AOAC (1990). Peroxide values were determined by the iodometric titration method in BS 684, Section 2.14 (1987). Saponification value, unsaponifiable matter and ash contents were analyzed according to the method described in Pearson's Chemical Analysis of Foods (Egan, Kirk, & Sawyer, 1981). Sugar was determined by extracting with ethanol–water (80/20, v/v) mixture at 80°C (Luh, Wong, & El-Shimi, 1981). The difference between 100 and the sum of the percentages of moisture, ash, protein, oil and sugar was reported as fibre content. For kernel to nut ratio determination, 10 g of whole pistachio nuts in triplicate were broken manually and carefully. They were separated from their shells and weighed separately. Then, the weight of kernel to total pistachio nuts weight was determined. Calorific value of pistachio nuts kernel was calculated by igniting in an adiabatic bomb calorimeter (Julius Peters k.g. Berlin 21) in triplicate.

2.5. Sensory evaluation

Sensory analysis was applied to determine quality of the pistachio nuts stored under different conditions. A taste panel of 8 panellists participated in the sensory evaluation of pistachio nuts. Panellists were experts from pistachio nut processing factories. Panellists scored pistachios by placing a vertical mark on an unstructured 10 cm horizontal line anchored at the ends by none (0) and extreme (10) intensity of the sensory characteristic.

2.6. Statistical analysis

The results were compared by one-way analysis of variance (one-way ANOVA) to test for significant differences. Means of the groups were compared using the least significant difference (LSD) multiple range test using a Statgraphics statistical packet (Statgraphics, 1991). Differences among sample means were reported to be significant when $p < 0.05$.

3. Results and discussion

The pistachio nuts was found to contain 3.98% water, 21.7% protein, 57.4% oil/fat, 13.5% sugar, 1.11% fibre and 2.31% ash. The results show that the pistachio nuts had a high lipid content. In our previous work, the fatty acid composition of pistachio nuts was determined and the unsaturated fatty acid content of the nut was found to be 89.8%, of the fatty acids present, 18.2% were polyunsaturated (Maskan & Karataş, 1998). The unsaturated fatty acid content makes it a nutritional product, but also makes it more susceptible to autoxidation. It is necessary to know the chemical and fatty acid composition of pistachio nut because of its relationship with nutrition and oxidation. In this study the composition of pistachio nuts is not the same as the values reported by Breuer (1993) Garcia et al. (1992) and Shokraii (1977). Percentage kernel (kernel/nut %) is one of the most important criteria which determine the nut quality. Our result (49%) is different from the data reported by Garcia et al. (1992), e.g. Antep, 53.5, Kellegouchi, 44.7 and Vahidi, 40.6. These differences are due to compositional differences between varieties. The calorific value of the pistachio nuts kernel was 660 kcal/100g of the edible part which is in agreement with those values reported by Breuer (1993) for tree nuts (cashews, 572, chestnuts, 194, raw peanuts, 571, roasted peanuts, 586, hazelnuts, 648, macadamias, 702, almonds, 598, brazil nuts, 666, pecans, 700, pine kernels, 674, pistachios, 642 and walnuts, 669 kcal/100 g edible part). The initial quality parameters of the pistachio nut oil were: FFA (% oleic acid), 0.363%, PV, 0.615, iodine value, 85, saponification value, 193 and unsaponified matter, 0.4%. Although chemical composition of pistachio nuts has been studied extensively, the oil characteristics have not. However, Danechrad (1974) has found that pistachio nut oil contained 0.54% unsaponifiable matter which is higher than our result. An overall comparison of pistachio nuts, considering the chemical composition and quality properties found for the vegetable oils reported by Swern (1964), shows that pistachio nut oil has similar characteristics (and composition) as olive oil except for its high oleic acid content.

Since hydroperoxides are the primary products of lipid oxidation, their content (i.e. peroxide value) is often used as an indicator for the initial stages of oxidation during storage. Fig. 1 shows the results of the PV against time at 10, 20, 30°C and ambient storages for pistachio nuts (each point represents the mean of two determinations).

It is seen from Fig. 1 that the differences in peroxide values due to storage conditions were not significantly different during 6 months of storage. After 6 months storage of pistachio nuts, the peroxide value of a sample stored under ambient conditions increased suddenly whole data were significantly different ($p < 0.05$) from that of samples stored under controlled conditions. These data show that peroxide values for the latter did not increase as fast as the former. The PV on ambient storage increased rapidly from 0.651 to 14.9, whereas PV of other conditions did not show a rapid increase. One-way ANOVA and LSD multiple range test results showed that the peroxide values at 10, 20 and 30°C under air and CO_2 storage were not different ($p > 0.05$), but significantly different from those under ambient storage conditions. The marked change in PV of samples stored under ambient conditions may be due to the seasonal variations in temperature and % RH of the atmosphere, which facilitate the activity of probable available lipoxygenase and lipase enzymes, light and presence of chlorophyll as a sensitizer (Bonvehi & Coll, 1993; Fakourelis, Lee, & Min, 1987; Labuza, 1971; Lopez et al., 1997a,b; Sowbhagya & Bhattacharya, 1976). Following autoxidation, particularly in samples stored under ambient conditions, the extracted oil was usually found to have become yellow, presumably showing lipid oxidation. The same observation was reported by Sowbhagya and Bhattacharya (1976) for lipid oxidation in rice and by Prior, Vadke, and Sosuiski (1991) for Canola oil. The effect of temperature on PV is apparent and expected. However, the rate of peroxide formation in samples stored under CO_2 was slower than air-storage at the same temperatures. Similar results were obtained by Waletzko and Labuza (1976) during storage of an intermediate moisture food in air and in an oxygen-free atmosphere and by Kacyn et al. (1983) for oxidation of a dehydrated model system containing methyl linoleate at low oxygen pressures. Holaday, Pearson, and Slay (1979) have also examined the quality of raw and roasted peanuts and pecans flushed with air or CO_2 and sealed in plastic pouches for long-term storage. They have found that the flavour and colour of the samples stored in the CO_2 atmosphere were better than those of the refrigerated controls and in air systems. In addition, they have reported that there was no evidence of insect activity or mold growth in any of the CO_2-treated samples.

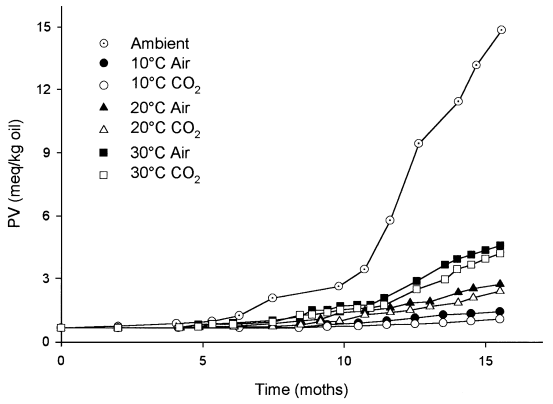

Fig. 1. Peroxide value of the whole-split pistachio nuts stored under various conditions.

3.1. Estimation of Arrhenius parameters

In order to estimate the Arrhenius parameters, the most common classic method-two steps least squares fit-was applied. In this method the non-linear regression of C versus t was performed at each temperature to estimate the rate constant k. For this, a computer package program (SigmaPlot Scientific Graphing System Version 4.10) was used. The peroxide value data of pistachio nuts from all storage conditions best fitted the first order rate kinetics model with correlation coefficients greater than 0.91. The reaction rate values obtained at 10, 20 and 30°C are plotted against reciprocal absolute temperature, T, and shown in Fig. 2. Each set of results was represented by Arrhenius-type relationships such as:

$$dC/dt = k(C) \quad (1)$$

$$k = k_o \exp(-Ea/RT) \quad (2)$$

where; C is peroxide concentration at time t, k is the rate constant (month^{-1}), k_o is pre-exponential factor, E_a is activation energy (kcal/mole), T is absolute temperature (°K) and R is the gas constant (1.98 kcal/mole.K).

The k value for each sample was calculated from Eq. (1), with reasonable correlation coefficients as shown in Table 1, and E_a and k_o values were estimated from slopes and intercepts of ln(k) versus $1/T$ plot (Fig. 2), respectively, by employing a linear least squares fit method. The reaction rate constants for the air system at 10, 20 and 30°C were found to be: 0.0686, 0.130, 0.182, respectively, and, for the CO_2 system at these temperatures: 0.0375, 0.123, 0.179, respectively. The reaction rate for the ambient system was also estimated as 0.254.

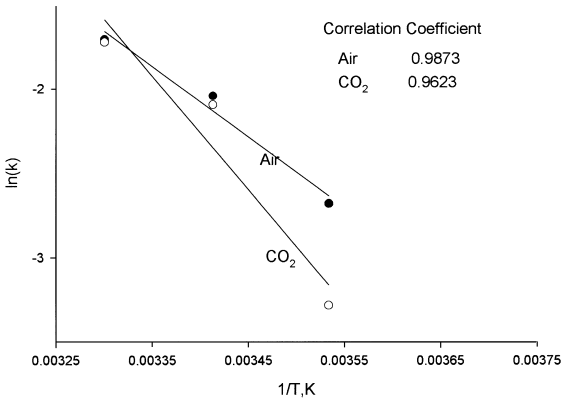

Fig. 2. Ln(k) versus $1/T$ plot for peroxide formation on the whole-split pistachio nuts stored in CO_2 and air.

Thus, in Fig. 2, the plots gave straight lines with correlation coefficients (r) of 0.9873 and 0.9623 for air and CO_2 systems, respectively. One would expect this plot to give a straight line for each system if the temperature does not change the mechanism. In addition, the lines should be parallel if there is not a change in mechanism. From this figure, it is clear that the plots of CO_2 and air systems are not parallel to each other. This suggests two different oxidation mechanisms (Labuza, 1982; Ragnarsson, Leick, & Labuza, 1977), in which the CO_2 system is different from the air system during oxidation. The plot of the CO_2 system is steeper than the air. This steeper slope of the CO_2 system means that the reaction occurring in the presence of CO_2 is more temperature-dependent/sensitive than air or, as temperature increases, even slightly, the reaction increases faster (Labuza, 1982).

Table 1 lists k values of peroxide formation at three temperatures of air and CO_2 and ambient systems. The k value of the ambient system is rather higher than those at 10, 20 and 30°C. This indicates that reaction rate under ambient conditions was about 3.70, 1.95, 1.39 times higher than that of the air system and 6.77, 2.05, 1.42 that with CO_2 storage at 10, 20 and 30°C, respectively. Also there are significant differences between k values of the CO_2 and air systems as shown in Table 1. As temperature increases this difference becomes negligible.

Possibly the reduced solubility of CO_2 in the oil and moisture portions of pistachio nut makes it ineffective as temperature increases. The solubility of the gas in water is inversely proportional to storage temperature and thus low temperatures have a synergistic effect upon its action as described by Church and Parsons (1995). The results at low temperatures, 10 or even 20°C, are in agreement with this statement and, at these temperatures,

Table 1
Estimated parameters from first order rate equation on peroxide formation of the whole-split pistachio nuts

	10°C		20°C		30°C		
	Air	CO_2	Air	CO_2	Air	CO_2	Ambient
C_0	0.481	0.552	0.369	0.340	0.2896	0.268	0.311
SE(±)	0.0464	0.0353	0.0310	0.0378	0.0302	0.0270	0.0854
k	0.0686	0.0375	0.130	0.123	0.182	0.179	0.254
SE(±)	0.0079	0.0056	0.0065	0.0086	0.0076	0.0074	0.0192
r	0.9512	0.9129	0.9883	0.9769	0.9907	0.9909	0.9874

CO_2 apparently has a protective effect on lipid oxidation. However, as temperature is raised to 30°C the effect of CO_2 decreases and the ratio of k values of air to CO_2 approaches 1. This means that there is no significant difference between storage in air and in CO_2 atmospheres. The unprotective effect of CO_2 at high temperature can be explained by the fact that the solubility of CO_2 in oil also decreases as temperature increases as mentioned by Swern (1964). In contrast to CO_2 the solubility of O_2, N_2, H_2 and CO gases in oil increases as temperature is increased. Availability of oxygen in storage atmosphere, even in small amounts at high temperature, increases the reaction rate, in spite of the presence of CO_2 in great amounts.

The experimental activation energies are the Arrhenius parameters derived from the experimental data, which describe the temperature dependence of reactant loss or product formed under those experimental conditions. The estimated activation energy values are 8.33 and 13.4 kcal/mole in air and in CO_2 atmosphere, respectively. Those Ea values for the CO_2 system are in reasonable agreement with the data presented for oxidation of a model food containing methyl linoleate, glycerol, water and microcrystalline cellulose in the presence of antioxidants which were from 9.0–22 kcal/mole (Ragnarsson et al., 1977). The high Ea value of the CO_2 system means that CO_2 has a reducing effect on lipid oxidation of pistachio nuts. The difference in Ea values of the air and CO_2 systems confirms the fact that CO_2 has a protective effect on lipid oxidation which correlates the findings of k values as shown in Table 1.

The change in fatty acid composition of pistachio nuts samples under the same storage conditions has been studied by Maskan and Karataş (1998).

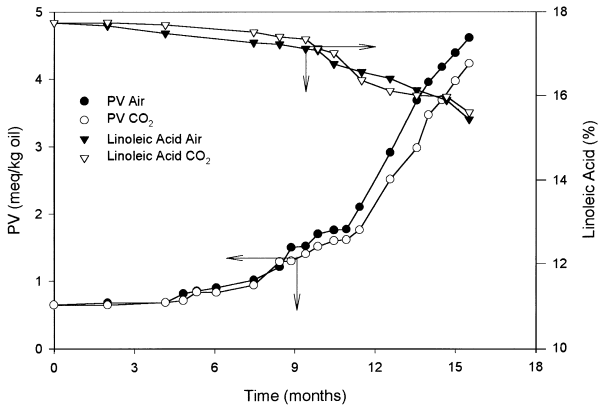

Fig. 3. Storage time of the whole-split pistachio nuts versus PV-Linoleic acid at 30°C.

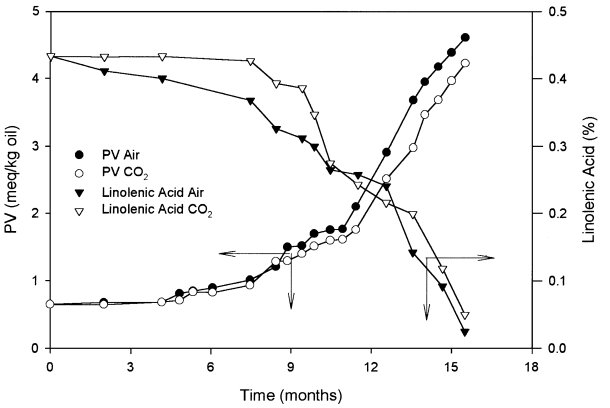

Fig. 4. Storage time of the whole-split pistachio nuts versus PV-Linolenic acid at 30°C.

Table 2
The results of LSD multiple range test for whole-split pistachio nuts quality parameters at $\alpha = 0.05$ level

Quality parameter	Storage type	Average	Homogeneous group[a]
Rancidity	Ambient	8.000	a
	10°C Air	0.750	b
	10°C CO_2	0.875	b
	20°C Air	1.000	b
	20°C CO_2	0.875	b
	30°C Air	1.250	b
	30°C CO_2	1.250	b
Overall flavour intensity	Ambient	3.500	c
	10°C Air	6.875	def
	10°C CO_2	7.625	ef
	20°C Air	7.000	def
	20°C CO_2	7.750	f
	30°C Air	6.500	de
	30°C CO_2	6.125	d
Colour	Ambient	3.000	g
	10°C Air	7.250	h
	10°C CO_2	7.000	ýh
	20°C Air	6.875	ýh
	20°C CO_2	6.875	ýh
	30°C Air	6.375	ý
	30°C CO_2	6.750	ýh

[a] Same letter indicates that difference is not significant at $\alpha = 0.05$ level.

However, it is interesting to make a relation between peroxide formation and fatty acid destruction for understanding the route of oxidation clearly. Our data showed that linolenic acid appears to be oxidized faster than linoleic acid under all conditions. The linolenic acid oxidized sooner and more rapidly than linoleic acid particularly under ambient conditions. The decrease in linolenic acid in both air and CO_2 storages at 20 and 30°C was relatively slow compared to ambient. However, both acids seem to be quite stable at 10°C and linoleic acid relatively stable at 20 and 30°C in air and CO_2. The fatty acids of pistachio nuts stored at M_o in the presence of air and CO_2 were relatively more stable than those under ambient conditions. It was observed that extensive reduction occurred in the two polyunsaturated fatty acids. The storage time versus linoleic/linolenic/PV at 30°C is presented in Figs. 3 and 4 as an example. Another interesting point here is that, in all cases, our data showed that the time at which the fatty acids decreased sharply nearly overlap with the time when PV increased (e.g., Figs. 3 and 4). The peroxide value increases rapidly during the propagation stage of the lipid oxidation free radical reaction. It has already been reported that, as tocopherols and pigments disappear, fatty acids begin to be oxidized and peroxide value increases (Prior et al., 1991; Rudolph, Odell, Hinrichs, Thomson, & Kays, 1992).

3.2. Sensory evaluation

The scores of panellists showed that the differences in crispness, moistness and oiliness of pistachio nuts are not significant ($p > 0.05$). However, the differences in rancidity, overall flavour intensity and colour is significant ($p < 0.05$) for the samples of different storages. LSD multiple range analysis showed that all samples are in a homogeneous group in terms of crispness, moistness and oiliness. But difference arises strongly due to rancidity, overall flavour intensity and colour disappearance for pistachio nuts stored under ambient conditions (Table 2). These results supports our previous findings and discussions of PV and fatty acid destruction.

4. Conclusion

The study of stability of whole-split pistachio nuts of Gaziantep variety revealed the following conclusions.

Fatty acid reduction and peroxide formation were more rapid in ambient storage. Storage at monolayer moisture content and in CO_2 improved the stability of pistachio nuts. However, as temperature increased the ratio of rate constants (air/CO_2) approached to 1 which means no significant difference between air and CO_2 storages. It was found that Ea value in CO_2 storage was greater than in air storage. This study could be useful for the storage of pistachio nuts, once hulled and split. The data provide interesting information on the effect of temperature and modified atmosphere on PV formation and fatty acid oxidation.

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