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Effect of Ripening of "Nabali" Olives on the Yield and Some Chemical Properties of Extracted Oil.

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ABSTRACT :

Olive samples were harvested every 2 weeks throughout the period from October, 1 to December, 31. The fresh samples were analyzed for moisture, oil content of the flesh and flesh/pit ratio. Oil samples which were extracted mechanically were stored at -18°C till the end of the experiment when they were analyzed for acidity, peroxide value, saponification number, iodine number and refractive index (at 25°C), as well as fatty acid composition using GLC.

Results indicated that the oil content of the flesh increased from 16.4 to 30.3%, whereas the flesh content of the fruits increased from 73 to 80% and the moisture content of the flesh decreased from 64 to 56% during the study. The acidity and the peroxide values, remained low and fairly constant (less than 0.33% and 2.35 respectively). Similarly, the values of the refractive index and saponfication number remained almost constant, whereas some variation in the iodine number was observed. There were some significant changes in the composition of individual fatty acids during the ripening process. Although these differences did not show a regular trend of change, archidic acid content in Nabali olive oil is shown to be higher than values reported for oil from other varieties; whereas stearic and linoleic acids were in the upper range reported.

Key words : Olive oil, Olive ripening, Nabali olives, Oil composition, Oil yield.

INTRODUCTION

Olive oil is a major agricultural product in Jordan. Most of the oil is produced from the Nabali variety which is favoured for its multi-purpose uses which include production of pickled green olives, ripe black olives and olive oil. It also has a relatively high oil content with an average of more than 20% (Al-Taher, 1947). It is generally recommended to delay harvesting of the fruits for oil production as this increases the oil yield, a fact which was reported by many workers (Snobar and Faqih, 1975; Ben Salah et al.; 1986, Marzouk and Cherif, 1981).

The influence of fruits ripening on the changes of quality of the extracted oil was also the subject of many studies. Catalano et al. (1975) determined the contents of neutral lipids and glycolipids of the olive fruits and leaves throughout the growing period. Marzouk and Cherif (1981) observed an increase of the polar lipids (phospho-and glycolipids) of the oil during ripening of the fruits. Ben Salah et al (1986) studied the evolution of lipids during over-ripening of the fruits (December to end of March). They observed many changes in the composition of lipids and fatty acids; one of which was the decrease in the relative proportion of linoleic acid and increase of the oleic acid in three olive varieties. Kiritsakis and Markakis (1984) monitored the changes in peroxide value (PV) and free fatty acid (FFA) content of the oil in relation to different collection regimes. They observed a significant increase in the PV and FFA in the fruits after harvesting but small and insignificant changes in these values when the olives remained on the trees.

The aim of this study was to investigate changes in fruit and oil composition of Nabali olives as well as some quality attributes of the extracted oil as related to the ripening of the fruits. This was thought to be important since no work has been published on this common variety in the area.

MATERIALS AND METHODS

Eleven olive trees of the Nabali variety were selected from trees in the University of Jordan campus at Jubeiha, Amman. Olive composite samples (5kg) were randomly hand-collected from the trees every 2 weeks starting October 1 and ending December 31, 1987. (Only the obviously unhealthy fruits were excluded).

On each collection day, a 200 g representative subsample was taken for the determination of moisture, fat content of flesh and flesh/pit ratio. The flesh was separated manually from the pits and both fractions were weighed. For moisture determination a flesh sample was dried to constant weight in an oven at 105°C (3-4 hrs.). The dried sample was then extracted with ether for 12 hrs. in a Soxhlet apparatus (AOAC, 1980).

For oil pressing, a 3kg subsample was passed in a hammer-type mill (Retsch Muhle, GmbH, 5657 Haan, West Germany) without using any of its sieves. The knives were adjusted so that the fruits were separated from the stones without breaking the pits. The pits were manually isolated from the disintegrated flesh. The flesh was pressed in cloth using a small hydraulic press. Oil was separated from the juice after warming it to 40°C using a laboratory centrifuge at 6000 rpm and then stored at -18°C till analyzed at the end of the experimental period.

Oil analysis which included determination of the acidity (expressed as % oleic acid), peroxide value, iodine number, saponification number and the refractive index at 25°C were carried out according to methods described by AOAC, 1980. The fatty acid composition was determined using a Hewlet Packard HP 5890A gas liquid chromatograph. A glass capillary column SP - 100 (30m x 0.25mm) was used (Temperature programme : 170 - 200°C, 1°C/min. det. FID 250°C; carrier gas (N_2) flow : 2.9 ml/min). The reference standard was a fatty acid methyl ester standard mixture (AOCS oil reference mixture, Supelco). Esterification of oil samples was done by sodium methoxide dissolved in benzene (Christie, 1976). The methyl esters were diluted with hexane. All analyses were done in triplicates.

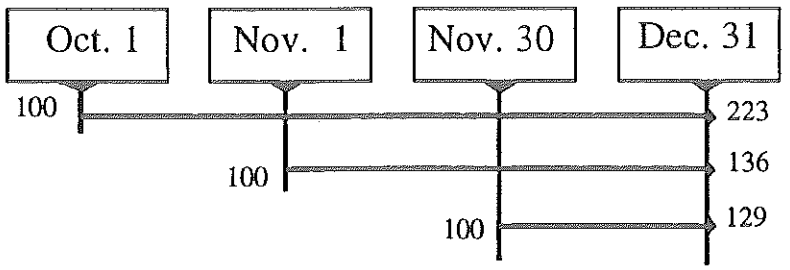

Fig. 1 Relative increase of oil yield in relation to harvesting date (calculated from changes in moisture, oil and flesh contents of the fruit) .

Data were statistically analyzed by the analysis of variance using Duncan's multiple range test (Little and Hills, 1972).

RESULTS AND DISCUSSION

Data in table 1 show the effect of ripening of olive fruits on their moisture and oil composition. It is obvious that the moisture content of the flesh gradually and significantly decreased throughout the study period; it dropped from 64.1% at the beginning of October to 56.1% at the end of December, i.e. 8% decrease. The oil content of the flesh increased in the same period by about 14% (from 16.4 to 30.3%); whereas the flesh proportion in the fruit increased from 79% to 83.5% as a result of fruit growth. Considering these changes, the expected increase in oil yield was calculated and the results are shown in figure 1. It is indicated that the delay of harvesting from the beginning of October to the end of December would give an overall increase of 223% in the oil yield. This yield would be 136% if harvesting was delayed from the beginning of November (the recommended date to start harvesting in Jordan) to the end of December (the actual end of the season). These figures support previous reports that ripening increases the yield of oil (Marzouk and Cherif, 1981; Ben Salah et al., 1986). However, the actual increase in the yield depends on many factors which affect the growth and drop of the fruits such as rain-fall, drought, wind, ambient temperature and health soundness of the fruits (Mustafa et al., 1987; Tapia, 1987).

In their study of lipogenesis during ripening of olives, Marzouk and Cherif (1981) described 3 phases : (1) A slow lipid biosynthesis phase in young recently formed fruits during which fruit growth occurs without any appreciable formation of lipids; (2) a rapid biosynthesis stage of oil in the fruits and (3) a stationary phase during which biosynthesis of lipids is slow or non-existing. The results listed in table 1 and figure 1 suggest that the selected observation period was falling within the second stage and that this stage had not finished by the end of December since the oil content increased over 6% in the last 2 weeks.

Table 1. Effect of time of harvesting on moisture, oil, flesh and pit contents in the whole olive fruits (% ± SEM).

Date of harvesting	% Moisture	% of oil in whole fruit	% of oil in flesh		% pit	% flesh
			fresh	dry malt		
October, 1	64.1 ± 0.6	13.6 ± 1.1	16.4 ± 1.0	45.7 ± 2.8	21.0 ± 1.1	79.0 ± 1.1
October, 15	59.4 ± 0.8**	15.8 ± 0.5	19.2 ± 0.6*	47.4 ± 2.0	21.9 ± 2.0	78.1 ± 2.0
October, 30	56.4 ± 0.2***	19.3 ± 1.2**	23.3 ± 1.2**	53.7 ± 2.8*	21.0 ± 0.6	79.0 ± 0.6
November,12	62.4 ± 1.0	18.7 ± 1.0*	21.8 ± 1.2*	59.1 ± 3.2**	16.3 ± 0.9*	83.7 ± 0.9*
November, 26	58.2 ± 0.8**	20.5 ± 1.2**	24.1 ± 1.5**	57.6 ± 3.6*	17.4 ± 1.1*	82.6 ± 1.1*
December, 14	55.6 ± 0.7***	20.2 ± 1.0**	23.8 ± 0.9**	55.0 ± 2.1*	17.6 ± 0.5*	82.4 ± 0.5*
December, 31	56.1 ± 0.6***	25.7 ± 1.6***	30.3 ± 2.0***	61.2 ± 4.0***	16.9 ± 1.0*	83.5 ± 1.0*

In each column
* indicates significant difference from initial value at ($p < 0.05$),
** indicates significant difference at ($p < 0.01$) and
*** indicates significant difference at ($p < 0.001$).

In table 2, the acidity and peroxide values of the oil samples obtained at different dates of the study are presented. The acidity, which is the most important chemical criterion for quality of virgin olive oil, remained low and almost constant (around 0.3%) throughout the observation period.

Table 2. Effect of time of harvesting of olives on the acidity and peroxide values of the extracted oil.

Date of harvesting	Acidity[1] (mean ± SEM)	Peroxide value[2] (mean ± SEM)
October, 1	0.30 ± 0.007	nd*
October, 15	0.31 ± 0.005	1.13 ± 0.08^{ac}
October, 30	0.30 ± 0.006	0.73 ± 0.06^{b}
November, 12	0.31 ± 0.010	0.77 ± 0.12^{b}
November, 26	0.32 ± 0.005	0.98 ± 0.08^{abc}
December, 14	0.29 ± 0.007	1.27 ± 0.09^{a}
December, 31	0.33 ± 0.003	0.83 ± 0.05^{bc}

1. Differences among acidity values were not significant.

2. Different letters indicate significant differences at $P < 0.01$.

* nd : not determined, sample lost.

According to the International Agreement on Olive Oil and Table Olives (UNCTAD, 1986), olive oil with acidity not exceeding 1.0% is considered to be "extra virgin oil". According to the Jordanian standards (Directorate of Standards and Measures, (1973), the acidity for "excellent" quality olive oil must not exceed 1.5%. The peroxide values showed significant differences among oil samples although they were all very low as compared with the maximum values

given for high-grade olive oil and thus the differences are not practically important. The observed fluctuation in the peroxide value could be due to the known instability of the hydro peroxides (Nawar, 1985). The relative stability of acidity and peroxide value is in agreement with results of other workers (Kiritsakis and Makakis, 1984) who reported that there were no significant changes in these two rancidity indicators within 60 days of observation. Furthermore, Ben Salah et al. (1986) stated that the free fatty acids in fresh overripe olives are only traces. It may be concluded that the lipase activity in the living fruits is very limited.

Data in table 3 show the iodine number, the saponification number and the refractive index of the oil samples. These tests which are used for the characterization of different oils were within the ranges reported for olive oil.

Table 3. Saponification number, iodine number and refractive index of olive oil obtained from olive fruits of different maturity levels (mean ± SEM).

Date of harvesting	Saponification number[1]	Iodine number[2]	Refractive index
October, 1	197 ± 0.6	80 ± 1.2[a]	1.4666
October, 15	196 ± 0.7	80 ± 0.7[a]	1.4668
October, 30	199 ± 0.6	85 ± 0.2[b]	1.4668
November, 12	198 ± 1.2	81 ± 1.2[a]	1.4668
November, 26	196 ± 1.2	81 ± 1.6[a]	1.4668
December, 14	193 ± 1.0	82 ± 0.7[a]	1.4668
December, 31	196 ± 0.5	88 ± 0.8[b]	1.4679

1. Difference among values of each column were not significant.

2. Different letters indicate significant differences at $P < 0.01$.

Table 4. Fatty acid composition of olive oil samples obtained at different ripening levels of the fruits (%+SEM).

Date of harvesting	Palmitic acid	Palmitoleic acid	Stearic acid	Oleic acid	Linoleic acid	Linolenic acid	Arachidic acid
October 1	10.2 ± 0.60^{bc}	1.1 ± 0.06^{a}	2.6 ± 0.06^{a}	75.6 ± 0.23^{c}	8.9 ± 0.06^{b}	0.4 ± 0.02^{b}	1.2 ± 0.02^{bc}
October 15	11.2 ± 0.10^{cd}	1.2 ± 0.06^{a}	2.5 ± 0.06^{a}	75.0 ± 0.30^{b}	8.5 ± 0.06^{b}	0.4 ± 0.06^{b}	1.2 ± 0.06^{bc}
October 30	11.8 ± 0.20^{d}	1.2 ± 0.07^{a}	2.9 ± 0.06^{a}	73.0 ± 0.30^{a}	9.6 ± 0.02^{d}	0.3 ± 0.06^{a}	1.1 ± 0.06^{b}
November 12	8.8 ± 0.10^{a}	1.5 ± 0.03^{bc}	3.0 ± 0.10^{a}	76.1 ± 0.20^{bc}	9.2 ± 0.06^{c}	0.5 ± 0.01^{c}	0.9 ± 0.01^{a}
November 26	9.5 ± 0.12^{ab}	1.6 ± 0.04^{c}	2.7 ± 0.06^{a}	75.0 ± 0.20^{bc}	9.6 ± 0.06^{d}	0.4 ± 0.06^{b}	1.1 ± 0.02^{b}
December 14	10.5 ± 0.20^{bc}	1.4 ± 0.04^{b}	2.8 ± 0.12^{a}	74.4 ± 0.20^{b}	9.3 ± 0.23^{c}	0.4 ± 0.02^{b}	1.1 ± 0.06^{b}
December 31	9.9 ± 0.20^{ac}	1.1 ± 0.06^{a}	2.6 ± 0.02^{a}	75.0 ± 0.27^{bc}	9.7 ± 0.02^{d}	0.4 ± 0.01^{b}	1.3 ± 0.02^{c}
Average	10.3	1.3	2.7	74.8	9.3	0.4	1.1

Different letters within each column indicate significant differences at $p < 0.01$.

The limited differences observed cannot be related to the fatty acid changes with the exception of the iodine number which might be attributed to the increase of linoleic acid as can be seen in table 4. The fatty acid composition of the oil samples agreed generally with those found in the literature with the exception of arachidic acid (C_2O : 0) which ranged from 0.9 to 1.3%. Arachidic acid values reported in the literature do not exceed 0.5% (Viola, 1964; Almirante et al., 1987). The presence of appreciable amounts of arachidic acid (1.2% as compared with "traces" in the literature) and stearic acid which had an average of 2.7% (reference range from 0.5 to 3.5%) may explain the generally observed higher viscosity of the local olive oil as compared with that produced in other countries such as Italy, Spain and Tunisia. Analysis of samples from 3 other locations, which were collected and analyzed for confirmation purpose supported this finding (table 5).

Table 5. Fatty acid composition (in %) of olive oil samples obtained from 3 different locations and the average of samples obtained from Jubeiha [1].

Fatty acid	Ajloun	Mahes	Nablus	Jubeiha samples (average)[1]
Palmitic acid	8.5	10.9	10.4	10.3
Palmitoleic acid	1.0	1.2	1.0	1.3
Stearic acid	4.0	2.9	4.6	2.7
Oleic acid	74.5	70.2	68.9	74.8
Linoleic acid	10.5	13.2	13.4	9.3
Linolenic acid	0.7	0.5	0.8	0.4
Arachidic acid	0.8	1.1	1.0	1.1

1. The Jubeiha values are the averages of values obtained at different dates of harvesting as in table 4.

An interesting observation regarding fatty acid composition was the significant increase in linoleic acid with ripening. Linoleic acid is an essential fatty acid which is recommended to supply not less than 1% of total daily caloric intake (Pipes, 1989). Other workers have reported variability in fatty acid content in relation to altitude (Lotti et al., 1982), to olive variety (Talantikite and Ait Amar, 1988) or to post-harvest conditions (Ben Salah et al., 1986). The present finding of increase in oil content as well as the increase in the essential fatty acid linoleic acid would suggest the importance of delaying the harvesting time of olives to get a better yield and quality of oil.

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دراسة أثر نضج الزيتون النباتي على كمية الزيت المستخرجة وبعض خصائصه الكيميائية

ملخص :

قطفت عينات من الثمار مرة كل إسبوعين في الفترة ما بين بداية تشرين أول (أكتوبر) ونهاية كانون أول (ديسمبر) ، وأجريت في كل مره تحاليل على عينه ممثلة للثمار مباشرة بعد القطف شملت نسبة الرطوبة ونسبة الزيت في اللحمية ونسبة اللحمية الى البذرة .

كما تم إستخراج الزيت من اللحمية بعصرها اليا . حفظت عينات الزيت ، بعد إستخراجها ، على - ١٨ م وحددت بعد إنتهاء فترة لتجربة نسبة الحموضة ورقم البيروكسيد ورقم التصبن والرقم اليودي ومعامل الإنكسار (على ٢٥ م) في العينات وكذلك حللت الأحماض الدهنية بالكروماتوغرافيا الغازية .

بينت النتائج أن نسبة الزيت في اللحمية تزداد م ٤ر١٦٪ الى ٣ر٣٠٪ ما بين بداية ونهاية التجربة (ثلاثة أشهر) في الوقت الذي إزدادت فيه نسبة اللحمية في الثمرة من ٧٣٪ الى ٨٠٪ بينما نقصت نسبة الرطوبة من ٦٤ الى ٥٦٪ .

أظهرت تحاليل الزيت أن قيم الحموضة ورقم البيروكسيد - وهما مؤشران للتزنخ - بقيت منخفضة وشبه ثابتة (أقل من ٣٣ر٠ ٪ للحموضة و ٣٥ر٢ لرقم البيروكسيد على التوالي) .

وكذلك بقيت قيم معامل الإنكسار ورقم التصبن ثابتة بينما كانت هناك بعض التغيرات المعنوية في الرقم اليودي أما الأحماض الدهنية فلم يكن هناك إتجاه واضح لتغيرها مع النضج على الرغم من حدوث تغيرات ذات دلالة معنوية لبعض الأحماض . وبنيت النتائج وجود إرتفاع في نسبة حمض الأراكيديك في الزيت المستخرج من الزيتون النباتي بالمقارنة بين نسبته في زيت المستخرج من الزيتون النباتي بالمقارنة بين نسبته في زيت الأصناف الأخرى والموجوده في المراجع ، ما كانت قيمتها حمضي الشمع (ستريك) والكتان (لينولينيك) مرتفعتين نسبيا .