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Kneading olive paste from unripe 'Picual' fruits: I. Effect on oil process yield

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ABSTRACT

Olive growers start earlier to harvesting in order to obtain high quality oils, however unripe olives show different characteristics affecting technological and rheological properties. This work is aimed to study the effect of malaxation conditions on process oil yield for unripe 'Picual' olives. Experiments were carried out using Picual fruits harvested at early dates for three consecutive crops seasons. The malaxation conditions assayed using a factorial design were: time (60, 90 min), temperature (18, 30, 40 °C) and micronised talc addition (0, 1%). Olive paste from unripe Picual fruit are classified as difficult pastes. The main factor affecting oil yield was the crop year, while for each crop year the effect of each factor varied considerably. Results indicated as increasing time and temperature gave lower pomace oil losses although its effect varied on crop year. Micronised Natural Talc addition lowered pomace oil content on dry weight. Therefore for early harvesting dates knowing olive characteristics 'on line' is essential to adapt the kneading conditions.

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1. Introduction

Virgin olive oil extraction is carried out by using only physical methods. Around 80% of oil located in the vacuoles of olive pulp cells can be easily extracted, the rest can be found dispersed in the cytoplasm as microgels and thus, is difficult to extract. In order to disrupt the interfacial films the olive 'paste is mixed slowly modifying the dispersion degree of oil droplets and promoting the coalescence phenomena. Coalescence increases the amount of free olive oil because of breaking of oil/water emulsion formed during the crushing operation. The oil droplets increase their size and when they achieve a diameter greater than 30 μm, the oil can be extracted easily (Martinez et al., 1957). Efficiency of kneading operation depends on olive paste rheological characteristics and malaxation variables such as time, temperature and technological coadjuvant addition.

Olive paste kneading conditions affect the process yield, improving when kneading time was prolonged from 50 to 75 min although kneading time excessively longer (105 min) did not give significant better results (Hermoso et al., 1998). Di Giovacchino (1991) described as olive paste kneading time higher than 45 min is needed to have good oil yields. Olive paste temperature decreases oil viscosity, improving the coalescence phenomena and the process yield (Hermoso et al., 1998; Di Giovacchino et al., 2002b). Extraction at 40 °C produces pomace with lower oil content than those obtained at 30 °C, although high temperature can affect negatively to oil quality and its sensory characteristics (Di Giovacchino, 1991; Hermoso et al., 1998).

Micronised Natural Talc (MNT) is used as technological coadjuvant because of its organophyllic properties reducing the oil/water emulsions and liberating oil for its extraction. Its use is recommended for the so-called 'difficult pastes' that show emulsions and low process yields (Martinez et al., 1975; Muñoz et al., 1979; Di Giovacchino, 1988). Experiments at industrial scale proposed the intermediate container of the paste kneader as the optimal location for talc addition since lower oil losses were observed. Different MNT doses have been tested, obtaining the best oil yields for 1%. When 2% of talc was added non-significant differences were observed although for difficult pastes, higher MNT amounts may be needed (Hermoso et al., 1998).

Most of these works were carried out just for a single date and crop year and they did not consider the effect of harvesting date and crop year for a specific harvest season. Furthermore some results are difficult to translate to other cultivars since extractability and paste rheological characteristics vary among cultivars (Beltran et al., 2003).

Picual is considered as the most important cultivar grown in Andalusia, Spain, EU and the World. In spite of its importance there are scarce data about its behaviour during oil extraction and how to process it. Among its cultivar characteristics, 'Picual' olive fruit falls on ground when ripens because of its low fruit retention force (Barranco et al., 2005). It occurs early, giving low quality oils from 'ground-picked' fruits. Because of that, may be interesting to anticipate the harvesting period in order to avoid oil quality losses. For

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the last years, in order to improve oil quality, sensory characteristics and nutritional composition more oil mills are extracting olive oil at earlier harvesting dates and lower temperature, however no data are available about the optimal kneading conditions using this kind of fruit to obtain rental oil yields. The aim of this work was to study how the malaxation conditions affect the oil process yield when early harvested fruit were processed.

2. Materials and methods

2.1. Plant material

This work has been carried out at the beginning of normal harvesting period for three consecutive crop years 2000/01, 2001/02 and 2002/03. Around 6000 kg of 'Picual' olive fruits were picked from mature trees grown under irrigation in the experimental olive orchard of IFAPA in Centro Venta del Llano in Mengibar, Jaén. For each year, trees were selected with a crop load index 3. Olives were harvested by a mechanical shocker and transported immediately to the pilot plant for processing. The harvesting dates and fruit ripeness index for each crop year are shown in Table 1.

2.2. Processing and extraction conditions

The olives were leaf-removal and not washed, an homogeneous lot of 6000 kg of olives was stored in a divided hopper with a common exit to improve the fruit homogeneity; batches of ~500 kg of olives were processed in each assay. Olives were crushed in a metallic hammer mill with a sieve size 6 mm for all the experiments, filling each batch a mixer container.

The IFAPA pilot plant was equipped with a thermobeater formed by three containers (Pieralisi, Spain), a two–three phases horizontal centrifuge Pieralisi SC-90 (working at two phases way) with a theorical processing capacity of 45,000 kg/day and a vertical centrifuge for oil cleaning Pieralisi P1500. The process was controlled and monitored by Procioleo automation software (Procisa, Seville). The olive paste was loaded at 750 kg/h in the horizontal centrifuge, being controlled by the automation system.

In the experiments three kneading temperatures were assayed (18, 30 and 40 °C) for two malaxation times (60 and 90 min). Micronised natural talc (MNT) was added at 1%, when used, by an automatic MNT dispenser previously calibrated. The experimental design is reported in Table 2.

Table 1
Harvesting date and ripening index for Picual olive fruit used in the malaxation experiments carried out during the crop years 2000/01, 2001/02 and 2002/03.

Crop year	Harvesting date	Ripening index
2000/01	11/29/00	2.82
2001/02	11/20/01	1.83
2002/03	11/27/02	2.42

Table 2
Factorial design of olive paste malaxation experiments.

t (min)	T (°C)	MNT (%)
60	18	0
		1
	30	0
		1
	40	0
		1
90	18	0
		1
	30	0
		1
	40	0
		1

2.3. Sampling

Olive samples were collected, by triplicate, at the beginning of each experiment of time and temperature being common for both MNT doses assays. Pomace samples were taken from the horizontal centrifuge outlet for the last 15 min of each assay in order to accurate the stabilisation of the decanter performance. Replicates were taken at 5 min intervals.

2.4. Analytical determinations

Fruit ripening index was measured according to the method the method proposed by the Estacion de Olivicultura y Elaiotecnia (Beltran et al., 2004). Crushed olives and pomaces (30 g) were weighted and dried until constant weight in an oven at 105 °C, the moisture was expressed as percent. Oil content was analysed by a Nuclear Magnetic Resonance (NMR) fat analyser mq 10NMR Analyser (Bruker, Spain) and were expressed as percent on fresh and dry weight basis. The equipment was calibrated periodically, for both fruit and pomace, by Soxhlet analyses using n-hexane as extracting solvent.

2.5. Statistical analysis

ANOVA analysis for a factorial design was performed using Statistix 8.0 Analytical Software in order to determine the effect of malaxation time, temperature and micronised natural talc on pomace oil content on dry weight. Tukey's test was applied to determine differences between means. Data for the treatment 18 °C 60 min and 1% of MNT of crop year 2001/2002 were missing.

3. Results and discussion

All the oils fitted within 'extra virgin' category as stated by European Union Regulation 2568/91 (1991). Quality parameters analysed were below the upper limits established for this category, the ranges observed for them were: Acidity (0.1–0.3%), Peroxide value (2.8–9.8 meqO_2 kg^{-1}), K_{232} (1.4–1.7) and K_{270} (0.11–0.21).

In Table 3 are reported the mean characteristics of olive fruits used in the experiments, as can be observed fruit characteristics varied significantly for each crop year affecting extractability and paste rheological properties. Fruits harvested during 2000/01 and 2002/03 crop years showed similar values for dry extract (%) and moisture (%), although for the last year lower oil content was registered probably because lipogenesis did not finish yet. Nevertheless the fruit with more important differences were observed in the crop year 2001/02 since they showed very high humidity (56.35%), high oil content (on dry weight) and very low level of dry extract. Because of this special characteristics, greater emulsions may be formed during crushing step giving difficult pastes.

Oil yield is usually expressed as pomace oil content on fresh weight (POFW), the main oil mill by-product, although when expressed on dry weight basis is not affected by the water addition in the process. Therefore results and discussion will be based on pomace oil content on dry weight (PODW). In Table 4 are reported the pomace oil content on dry weight for this work.

Considering the three crop years together, the ANOVA analysis (Table 5) showed as the crop year was the most important factor affecting the oil yield process (81.6%) whereas the malaxation variables explained around 2% of the observed variability. As shown in Table 4, oil yields were better for the crop year 2000/01 while the

Table 3
Characteristics of olive fruits used in the malaxation experiments carried out during the crop years 2000/01, 2001/02 and 2002/03.

Crop year	t (min)	T (°C)	MNT (%)	Moisture (%)	Oil/FW (%)[a]	Oil/DW (%)[b]	Dry extract (%)
2000/01	60	18	0	55.09	21.04	46.85	23.87
2000/01	60	30	0	50.81	21.48	43.67	27.71
2000/01	60	40	0	50.32	22.20	44.69	27.48
2000/01	90	18	0	53.95	20.74	45.04	25.31
2000/01	90	30	0	51.87	21.73	45.15	26.40
2000/01	90	40	0	50.79	20.92	42.51	28.29
2001/02	60	18	0	55.52	19.66	44.20	24.82
2001/02	60	30	0	57.94	19.96	47.46	22.10
2001/02	60	40	0	57.34	20.76	48.66	21.90
2001/02	90	18	0	57.00	19.20	44.65	23.80
2001/02	90	30	0	56.96	20.99	48.77	22.05
2001/02	90	40	0	53.34	20.57	44.08	26.09
2002/03	60	18	0	52.33	16.62	34.86	31.05
2002/03	60	30	0	53.27	19.82	42.41	26.91
2002/03	60	40	0	52.98	18.35	39.03	28.67
2002/03	90	18	0	52.86	18.11	38.42	29.03
2002/03	90	30	0	53.68	20.33	43.89	25.99
2002/03	90	40	0	54.60	19.55	43.06	25.85

[a] Oil content on fresh weight.
[b] Oil content on dry weight.

Table 4
Effect of malaxation conditions on pomace oil content on dry weight (%) for three consecutive crop years: 2000/01, 2001/02 and 2002/03.

T (°C)	t (min)	MNT (%)	Crop year			Mean
			2000/01	2001/02	2002/03	
18	60	0	7.39 ± 0.44[a]	10.64 ± 0.31	8.33 ± 0.34	8.79 ± 1.48
		1	7.17 ± 0.22		7.35 ± 0.41	8.25 ± 1.50
	90	0	6.36 ± 0.31	11.49 ± 0.51	7.75 ± 0.53	8.53 ± 2.33
		1	5.95 ± 0.17	10.67 ± 0.28	6.81 ± 0.53	7.81 ± 2.20
30	60	0	6.14 ± 0.33	12.24 ± 0.54	8.08 ± 0.77	8.82 ± 2.75
		1	5.78 ± 0.09	10.95 ± 0.98	7.76 ± 0.57	8.16 ± 2.33
	90	0	5.30 ± 0.08	10.10 ± 1.26	7.10 ± 0.19	7.50 ± 2.19
		1	5.19 ± 0.12	10.86 ± 0.34	7.20 ± 0.41	7.75 ± 2.51
40	60	0	5.69 ± 0.06	11.19 ± 0.43	7.49 ± 0.41	8.12 ± 2.45
		1	5.38 ± 0.09	11.05 ± 0.67	7.29 ± 0.30	7.91 ± 0.52
	90	0	5.99 ± 0.08	9.45 ± 0.24	7.30 ± 0.13	7.58 ± 1.52
		1	5.97 ± 0.20	8.22 ± 0.11	6.83 ± 0.34	7.01 ± 1.01

[a] Mean values ± SD (n = 3).

Table 5
Partial mean squares from analysis of variance for the effect of malaxation conditions on pomace oil content on dry weight (%) at early harvesting date for the crop years: 2000/01, 2001/02 and 2002/03.

Source	DF[a]	MS[b]	SST_0[c]	P
Total	107			
Crop year (C)	2	393.024	84.63	0.0000
Micronised talc (MNT)	1	4.535	0.98	0.0000
Temperature (T)	2	8.683	1.87	0.0000
Time (t)	1	11.258	2.42	0.0000
$C *$ MNT	2	0.413	0.09	0.3509
$C * T$	4	8.158	1.76	0.0000
$C * t$	2	1.036	0.22	0.0763
MNT $* T$	2	0.818	0.18	0.1291
MNT $* t$	1	0.098	0.02	0.4802
$T * t$	2	1.295	0.28	0.0412
$C *$ MNT $* T$	4	0.678	0.15	0.4848
$C *$ MNT $* t$	2	0.018	0.04	0.9547
$C * T * t$	4	15.992	3.44	0.0000
MNT $* T * t$	2	2.131	0.46	0.0061
$C *$ MNT $* T * t$	4	2.262	0.48	0.0272
Error	72	13.980	3.01	

[a] Degrees of freedom.
[b] Mean squares.
[c] Partial mean square for the effect, expressed as percentage, of the total corrected sum of squares.

worst were registered for 2001/02. This different extraction efficiency may be explained because of the large differences in fruit moisture recorded for the three crop years (Table 3). In general, olive pastes showing moisture higher than 50% are classified as difficult pastes (Di Giovacchino, 1991). Furthermore olive paste from crop year 2001/02 may be classified as 'very difficult paste', since it showed the highest moisture (56.3%), high oil content and a very low dry extract percent. During its processing the oil lost was very high (10%) and malaxation conditions did not improve significantly the oil yield. Therefore and because of the significant effect of crop year, a separated ANOVA analysis for each one was necessary to get more robust information (Table 6).

Malaxation temperature affected the process oil yield although its effect varied significantly depending on the fruit characteristics for each year (Fig. 1). The low oil yield improvement obtained when temperature was increased is due to the difficult pastes produced from these olive fruits. For crop year 2001/02 only for 40 °C a significant reduction in the oil losses was observed whereas for 2000/01 crop year temperature was the main factor affecting the PODW; non-significant effect was observed for 2002/03 crop year. Increasing malaxation temperature reduced the pomace oil content on dry weight for all the crop years studied, obtaining the lower values and thus, the highest yields, kneading at 40 °C. But when similar temperature increase was performed we did not observed

Table 6
Partial mean squares from analysis of variance for the effect of malaxation conditions on pomace oil content on dry weight (%) at early harvesting date for the crop years: 2000/01, 2001/02 and 2002/03.

Crop year	DF[a]	Total	MNT	T	t	$T*t$	MNT $*T$	MNT $*t$	Error
		35	1	2	1	2	1	2	26
2000/01	MS[b]	16.506	0.509**	8.791***	1.960***	3.963***	0.028	0.035	1.223
	SST_0[c]		3.08	53.25	11.87	24	0.17	0.21	7.41
2001/02	MS	35.642	2.704*	3.367**	3.555*	4.911***	0.305	0.353	0.516
	SST_0		7.59	18.89	9.97	27.56	1.71	0.99	33.28
2002/03	MS	11.899	1.979**	0.412	2.745***	0.151	0.577	0.009	0.196
	SST_0		16.63	6.92	23.07	2.54	9.70	0.07	41.07

[a] Degrees of freedom.
[b] Mean squares.
[c] Partial mean square for the effect, expressed as percentage, of the total corrected sum of squares.
* Significance level p: 0.05.
** Significance level p: 0.01.
*** Significance level p: 0.001.

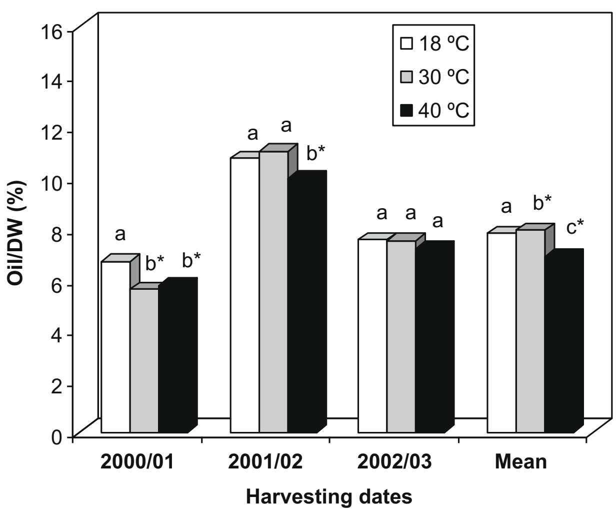

Fig. 1. Effect of malaxation temperature on pomace oil content on dry weight for three consecutive crop years: 2000/01, 2001/02 and 2002/03. Different letters mean significant differences at *p: 0.05, **p: 0.01 and ***p: 0.001.

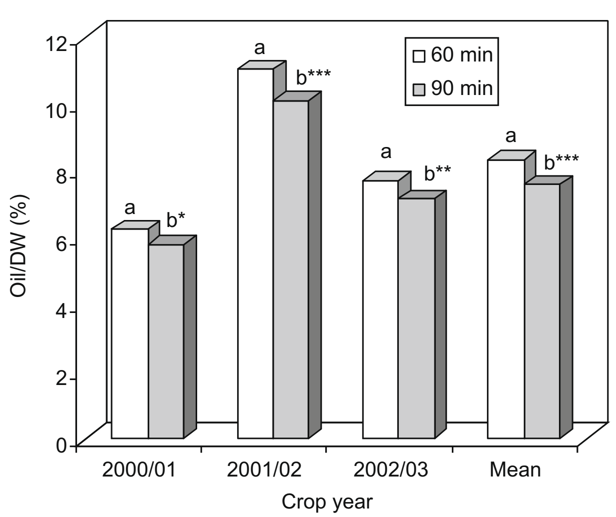

Fig. 2. Effect of malaxation time on pomace oil content on dry weight for three consecutive crop years: 2000/01, 2001/02 and 2002/03. Different letters mean significant differences at *p: 0.05, **p: 0.01 and ***p: 0.001.

the same oil yield reduction. For the first crop year higher temperature than 18 °C improve the process yield significantly by 16.7%, however for the second one only increasing malaxation temperature to 40 °C let to reduce the pomace oil losses. Although, in general, a decrease of PODW was observed for higher malaxation temperature, attention should be paid into the values obtained for 18 °C since only for one crop year oil yield were worse than 30 °C and thus, it may be interesting to process at low temperature to obtain Virgin olive oils with singular characteristics.

Longer malaxation times gave significant lower PODW (Fig. 2), as described previously (Hermoso et al., 1998; Di Giovacchino et al., 2002a), since increasing kneading time let to reduce the emulsion formed during crushing operation, increasing the 'free oil' in the olive paste. This variable achieved higher importance for the crop year 2002/03 (Table 6) although was significant for all the crop years studied. Prolonging malaxation from 60 to 90 min gave similar reduction in PODW for the three crop years: around 7%, independently of the fruit characteristics.

Although both, time and temperature showed a significant effect on oil yield by themselves, their interaction showed a highly significant effect for the crop years 2000/01 and 2001/02 (Table 6). In Fig. 3 interaction between time and temperature is represented for each crop year. For 2000/01 a significant decrease in PODW was observed only when kneading at 18 °C for 90 min. However for the crop year 2001/02, the significance of this interaction was higher as the temperature rose: malaxing at 30 °C for 90 min lowered the PODW by 9.7% whereas this reduction was 20.5% at 40 °C. The important effect of this interaction may be explained because olive paste during 2001/02 can be classified as very difficult paste (moisture higher than 56%) and thus both higher temperature and longer time are needed to reduce the microgels and semicolloidal structures described for these olive pastes.

The third variable studied was the addition of micronised natural talc (MNT) as technological coadjuvant. MNT lowered significantly (Fig. 4) the pomace oil content on dry weight ($p < 0.05$) as described for 1% MNT addition by Hermoso et al. (1998), this reduction varied depending on the fruit characteristics for each crop year. MNT was more efficient for crop year 2002/03 reducing the oil lost by 8.1% whereas for 2000/01 that reduction was lower since acceptable oil yields were obtained for all the treatments. Although crop year 2001/02 had very difficult pastes, MNT reduced the oil losses by 6.1%, the high oil losses indicated that higher MNT should have been added.

Processing unripe Picual fruits required adapted processing conditions since because of their own characteristics all the olive pastes can be considered as 'difficult' even 'very difficult' pastes. In general acceptable oil yields were obtained under the malaxation conditions assayed since values of pomace oil losses were lower than those described previously for two phases system (Di Giovacchino et al., 2002a; Inarejos-Garcia et al., 2009). In general,

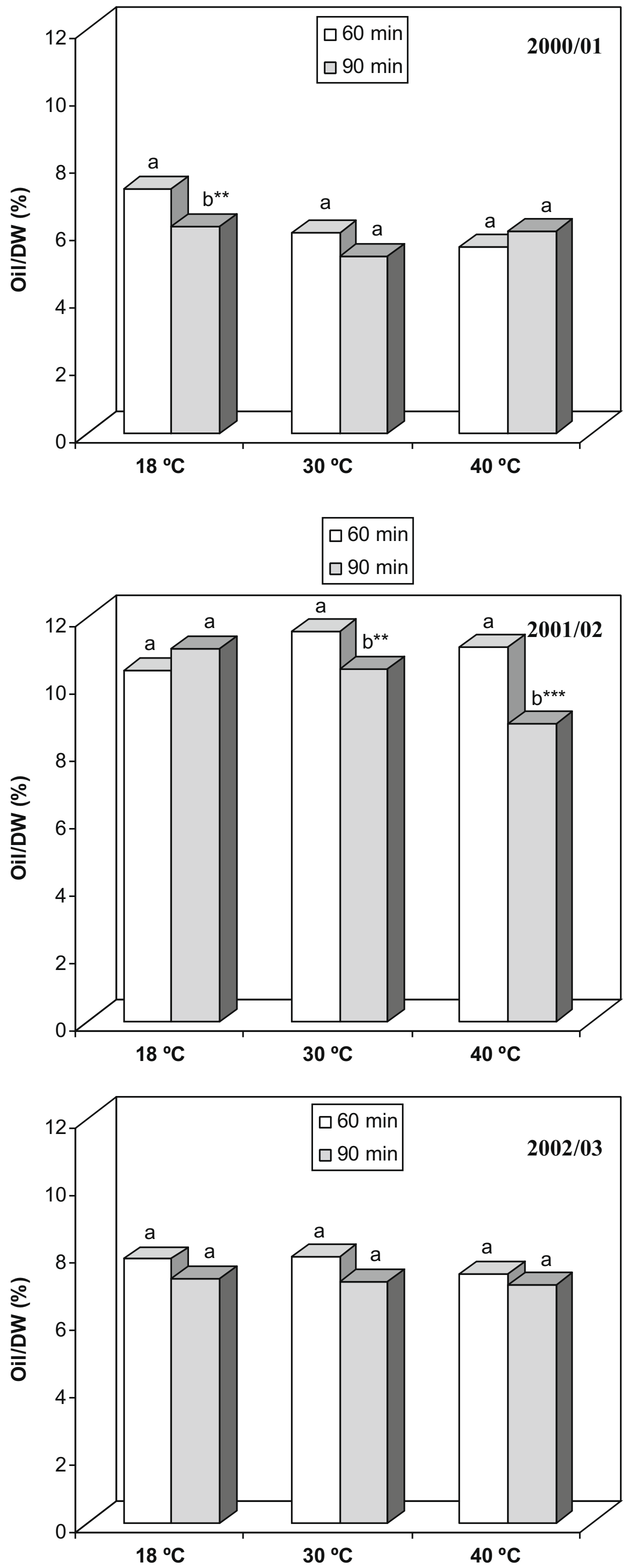

Fig. 3. Interaction between malaxation time and temperature for pomace oil content on dry weight from early harvesting fruits for three consecutive crop years: 2000/01, 2001/02 and 2002/03. Different letters mean significant differences at $^{*}p$: 0.05, $^{**}p$: 0.01 and $^{***}p$: 0.001.

temperature let to improve their oil yields just for 40 °C than can have negative effect on oil quality. Longer malaxation reduced by 7% the pomace oil content while MNT addition (1%) lowered it

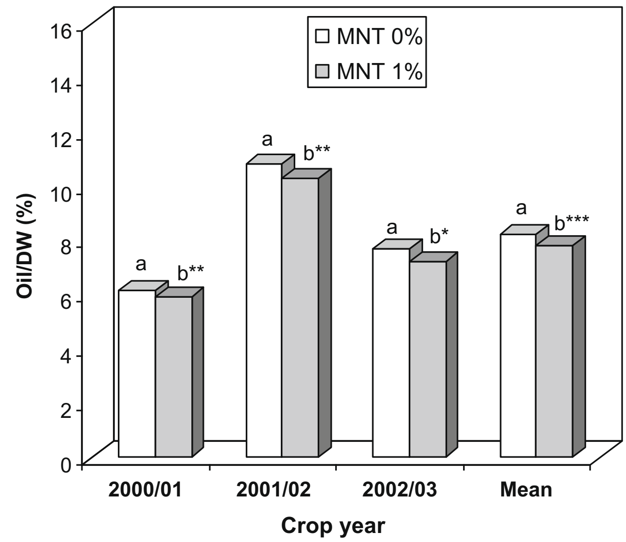

Fig. 4. Effect of Micronised Natural Talc (MNT) addition on pomace oil content on dry weight for three consecutive crop years: 2000/01, 2001/02 and 2002/03. Different letters mean significant differences at $^{*}p$: 0.05, $^{**}p$: 0.01 and $^{***}p$: 0.001.

by 6.13%. Considering the worst paste rheological conditions, moisture ∼56%, for each temperature the oil yield was improved by both longer malaxation time and MNT addition. The different malaxation conditions required for each fruit characteristics, makes necessary the development of potent tools for olive fruit characterisation 'on line' in order to adapt the process to the olives.

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