

Research Article

Virgin olive oil yield as affected by physicochemical talc properties and dosage

Abir Sadkaoui¹, Antonio Jiménez¹, Maria P. Aguilera², Rafael Pacheco³ and Gabriel Beltrán¹

¹ IFAPA Center “Venta del Llano”. Junta de Andalucía. Ctra. Bailén-Motril, Mengíbar (Jaén), Spain

² Technological Center of Olive Grove and Oil, Citoliva Foundation, Scientific-Technological Park GEOLIT, Mengíbar, Jaén, Spain

³ Department of Chemical, Environmental, and Material Engineering, University of Jaén, Jaén, Spain

The aim of this work was to assess the effect of physicochemical characteristics of micronized natural talc (MNT) and its dosage on the industrial oil yield (IY) during virgin olive oil (VOO) extraction of Hojiblanca fruits. For this purpose, laboratory scale experiments were performed testing four commercial MNTs differing in their particle size (D50) and carbonate rate at doses ranging from 0 to 3%. The most efficient MNT was characterized by the highest carbonate rate and the lowest particle size achieving the highest improvement of the IY for a dose of 2%. It was also found that the effect of particles size and carbonate rate depends on the MNT dose, being much more pronounced at lower doses and that the use of MNTs with low carbonate levels or high particle size could be compensated using higher doses of talc.

Practical applications: This study provides deeper insights into the clarification of the effect of the addition of different doses of MNT, with different physicochemical characteristics on oil yield of Hojiblanca olive fruits. The information contained in this study should be taken into account to choose the suitable MNT dose considering its physicochemical characteristics in order to improve the oil yield for the so-called difficult pastes.

Keywords: Carbonate rate / Micronized natural talc / Particle size / Process industrial yield / Talc dose

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1 Introduction

Virgin olive oil (VOO) is extracted from olive fruit using only mechanical processes including olives crushing, malaxation

of resulting pastes, and separation of the oily phase essentially by pressure or centrifugation [1].

Oil contained within the mesocarp cells is released during the crushing and malaxing process. In some cases, oil is retained within the cell walls rather than being released during the crushing and malaxation [2]. Such pastes are called fluent or difficult, and are typically obtained from varieties as the Spanish Hojiblanca and Picual, and occasionally from other varieties when freshly harvested olives are processed [3]. Among the factors affecting to extractability of the olive paste can be considered the pectic substances derived from cell wall polysaccharides. These substances are natural emulsifier agents present in olives. They promote emulsion formation and short-term stabilization by interfacial action [4] as confirmed by Sadkaoui et al. [5] who established that increasing pectin concentration gave a rise in the emulsifying activity. These emulsions are surrounded by lipoproteic membranes preventing the merging of the oil droplets spread in the malaxed olive

Correspondence: Abir Sadkaoui, IFAPA Centro “Venta del Llano,” Junta de Andalucía, P.O. Box 50, Mengíbar, Jaén E-23620, Spain

E-mail: abir_sadkaoui@yahoo.fr

Fax: +33 1 47 99 54 90

Abbreviations: ANOVA, analysis of variance; **D 50**, particle size diameter; **DF**, degree of freedom; **FW**, fruit weight; **IY**, process industrial yield; **M**, moisture content; **MNT**, micronized natural talc; **MS**, mean squares; **OFDW**, fruit oil content on dry weight basis; **OFFW**, fruit oil content on fresh weight basis; **P**, statistical significance; **r²**, coefficient of determination; **RI**, ripening index; **S_{BET}**, specific surface area; **SD**, standard error; **SST₀**, partial mean square for the effect, expressed as percentage of the total corrected sum of squares; **SW**, stone weight; **T**, temperature; **X_{carb}**, percentage of carbonate content in the talc

paste [6]. This phenomenon eventually leads to the production of intermediate phases of very similar density to oil and water difficult to separate and then, affecting the oil mill working capacity and increasing the oil losses in the pomace [7]. In order to break these emulsions and improve the oil yield, different strategies have been successfully developed such as increasing time and temperature of the malaxation and using technological coadjuvants. Regarding the use of technological coadjuvants, a number of trials proved that the addition of enzymes during malaxation step determines an increase in oil extraction yield [8, 9]. However, the use of technological coadjuvants [3] with chemical or biochemical activity is forbidden by European regulations on VOO production [10]. The use of talc powder [11–13], salt [14, 15], and calcium carbonate [16] has been proposed as physical acting coadjuvants alternative to enzymes. Actually, micronized natural talc, the natural form of the hydrated magnesium silicate, $Mg_3(Si_4O_{10})(OH)_2$, is the most widely used coadjuvant for oil extraction [11, 13] due to its exclusively physical action [17]. According to Caponio et al. [18], this mineral is widely used due to its intrinsic properties such as chemical inertness [19]. Talc shows both hydrophilic and hydrophobic behavior at its breaking surfaces. After milling, charged and non-charged surfaces are formed as a consequence of particle breaking [20]. This mineral has been found to destabilize emulsions making solid/liquid separation easier and increases the oil yield; reduces notably the amount of fine solids in the oil and does not produce any changes in physical, chemical, or organoleptic properties of the oil [2]. At the end of the extraction process, the talc ends up in the olive pomace, because of its specific weight (2.8 g/cm³), while the oil is free of this substance [18]. However, because talc is a natural product, its physicochemical characteristics vary depending on the deposit and the production location. Each talc mine has its own characteristics and that is why talc grades differ in their performance in the end applications. Among the most studied MNT characteristics, particles size and carbonate rate may affect substantially the industrial oil yield [21–23].

The current work was carried out under controlled laboratory scale conditions in order to gain a better understanding of the effect of different doses of MNTs with different physicochemical characteristics on the industrial oil yield during VOO extraction of Hojiblanca fruits.

2 Materials and methods

2.1 Raw material

Olive trees (*Olea europaea*, L.) of Hojiblanca cultivar with a similar crop load were selected. The trees were spaced 7 × 7 m and grown in the experimental orchard of IFAPA “Venta Del Llano” Mengíbar, Jaén (Spain) using standard growing techniques. The study was performed during 2009/

10 crop year. A lot of 200 kg of olives was harvested by hand, homogenized, cleaned, and processed immediately. A total of 100 fruits were taken at random in order to determine their fresh weight, stone weight, and their ripening index determined according to the method described by Uceda and Frías [24].

Some fruits were destoned and the flesh (pulp) was separated. Fruit and flesh were crushed and 60 g of olive paste were dried in a forced air oven at 105°C for 24 h. The dried paste was weighed and the moisture content recorded. The oil content in the fruit was determined using a Nuclear Magnetic Resonance (NMR) fat analyzer mq 10NMR (Bruker, Spain) and expressed as a percentage on both a fresh and a dry weight basis. The equipment was calibrated periodically by Soxhlet analyses using n-hexane as extracting solvent. Characteristics of the olive fruit used for the experiment are given in Table 1. All the analyzed parameters were those normally detected in olives harvested at a ripening index of 4.5.

Four commercial MNTs have been used; their main physical and chemical characteristics are shown in Table 2.

2.2 Processing and olive oil extraction conditions

Experiments were performed at laboratory scale, using Abencor extraction unit (Abencor series 100, MC2 ingeniería y Sistemas, S.L. Seville, Spain), simulating commercial oil extraction systems. A total of 64 samples (16 treatments × 4 replicates) were processed. Olives were crushed using a 6 mm sieve diameter. The olive paste was then malaxed in a thermo-beater at 28°C for 45 min. In the experiment, a factorial design was selected considering two variables: type of MNT (MNT1; MNT2; MNT3; and MNT4) and MNT doses (0, 1, 2, and 3%). Four replicates were carried out for each treatment. The MNT, when applied, was added at the beginning of malaxation.

The process industrial yield (IY) was defined as the percentage of oil obtained using the Abencor laboratory oil extraction system from 700 to 800 g of olives paste. The volume of olive oil was measured on graduated cylinder and the oil yield was calculated using the olive oil density of 0.915 kg/L Eq. (1). Results were expressed as percent.

$$IY = \left[\frac{(OV \times 0.915)}{WOP} \right] \times 100, \quad (1)$$

Table 1. Initial composition of Hojiblanca olive fruit employed in the technological assays

RI	M (%)	OFFW (%)	OFDW (%)	FW (g)	SW (g)
4.5	53.87	20.45	44.34	3.70	0.66

RI, ripening index; M, moisture content; OFFW, fruit oil content on fresh weight; OFDW, fruit oil content on dry weight; FW, fruit fresh weight; SW, stone weight.

Table 2. Basic data of the micronized natural talc (MNT) used in the experiment

Coadjuvant	Carbonate (%)	Medium particle size D50 (μm)	Density (g/cm ³)	Specific surface B.E.T (m ² /g)
MNT 1	4	2.4	2.77	7
MNT 2	0.8	7.1	2.78	9
MNT 3	0.5	13	2.77	7
MNT 4	1.1	16.3	2.78	6

B.E.T, Brunauer, Emmett and Teller method is the most widely used procedure for the determination of the surface area of solid materials.

where OV is the volume of olive oil obtained from the Abencor system (L) and WOP is the weight of olive fruit paste used (kg).

2.3 Statistical analysis

In the tables, results are expressed as mean values ± standard deviation (SD). Analysis of variance (ANOVA) was applied. Tukey's HSD test was used to determine significant differences between means ($p \leq 0.05$). These determinations were carried out using the software Statistix, Version 9.0.

3 Results and discussion

From the ANOVA (Table 3) was observed as both MNT dose and MNT type showed a very level of significance on the process IY. However, MNT dose could explain the highest percent of variability (55%) over the value achieved by MNT type (12.4%). Interaction between both factors was not significant. These results agreed with previous works [2, 22].

In general, the addition of MNT improved the oil yield in all cases when compared to control without MNT addition (Table 4). These results agree with previous works [12, 25,

Table 3. Partial mean squares from analysis for the effect of talc physicochemical characteristics and dosage on the process industrial yield (IY) of 'Hojiblanca' fruits

	Total	MNT (T)	Dose (D)	T × D	Error
DF ^a	63	3	3	9	48
MS ^b		6.47	28.62	1.12	0.86
SST ₀ ^c		12.39	54.81	6.46	26.34
P ^d		0.0003	0.0000	0.2579	

^aDegree of freedom.

^bMean squares.

^cPartial mean square for the effect expressed as percentage of the total corrected sum of square.

^dStatistical significance.

Table 4. Effect of MNT dose and physicochemical characteristics on the process industrial yield (IY)

MNT	Carbonate (%)	D 50 (μm)	MTN (%)	Oil yield (%)	Improvement respect to the control (%)
MTN 1	4	2.4	0	8.34 ± 0.40	–
			1	11.27 ± 0.56	26
			2	11.84 ± 0.03	29.56
MTN 2	0.8	7.1	0	8.34 ± 0.95	–
			1	9.08 ± 1.02	8.15
			2	10.77 ± 1.00	22.56
MTN 3	0.5	13	0	7.49 ± 0.48	–
			1	8.86 ± 0.33	15.46
			2	10 ± 0.33	25.1
MTN 4	1.1	16.3	0	8.42 ± 0.28	–
			1	9.42 ± 0.32	10.62
			2	10.99 ± 0.35	23.38
			3	10.98 ± 0.86	23.32

D 50, particle size; MNT, micronized natural talc; MNT 1, (4% (CaCO₃); 2.4 mm); MNT 2, (0.8% (CaCO₃); 7.1 mm); MNT 3, (0.5% (CaCO₃); 13 mm); MNT 4, (1.1% (CaCO₃); 16.3 mm).

26] that reported higher yields when MNT was used. The capacity of talc to increase oil yields could be explained by its exclusively physical action since it adsorbs the droplets of oil retained in the cell walls, forming larger drops, thereby facilitating the oil extraction [6]. In the olive paste, oil and water can get entrapped in agglomerating microgels. Additionally, both olive paste and talc have lipophilic characteristics, therefore, they tend to combine with or retain the oil. The talc helps counteract the paste further releasing oil into an extractable form, breaks up these microgels and release the oil [27]. It also prevent the formation of oil-in-water emulsions and breaks up emulsions previously formed, allowing the oil to be extracted [5, 28, 29].

Because of the higher effect of MNT dose on oil yield, MNT dose effect was analyzed for each MNT independently. It could be observed as the four MNTs behaved differently depending on the dose applied to the olive paste (Table 4). The highest IY values were obtained for MNT1 which produced a lineal increase of the IY as talc was higher. The lowest values were obtained when MNT3 was added at doses ranging between 1 and 2%. Similar trend was observed for the trials carried out with MNT2 and MNT4 only up to the dose of 2% beyond which either a stabilization (MNT 4) or a slight decrease of the IY (MNT 2) was observed.

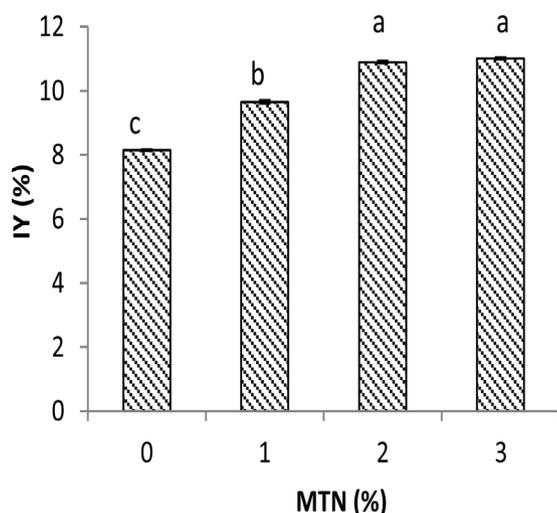


Figure 1. Effect of the MNT dose on the process industrial yield (IY) of Hojiblanca fruits considering the four MNTs together. Different letters means significant differences at p , 0.05.

Considering the pool of four MNTs together (Fig. 1), the addition of MNT at doses ranging between 1 and 2% gave a rise in the IY, while greater doses (3%) gave any significant variation on the IY. The doses for talc addition referred in the literature for olive oil extraction range between 0.5 and 2% [28, 29]. However, excessive doses can have a negative effect on the extraction process [28].

In general, at lower doses (1 and 2%), MNT1 showed to be the best coadjuvant in terms of extraction efficiency while MNT3 gave the worse results (Table 4). Considering the pool of MNT doses, among the MNT assayed can be observed as MNT 1 gave the highest oil yield achieving significant differences respect to MNT2 and MNT3 (Fig. 2).

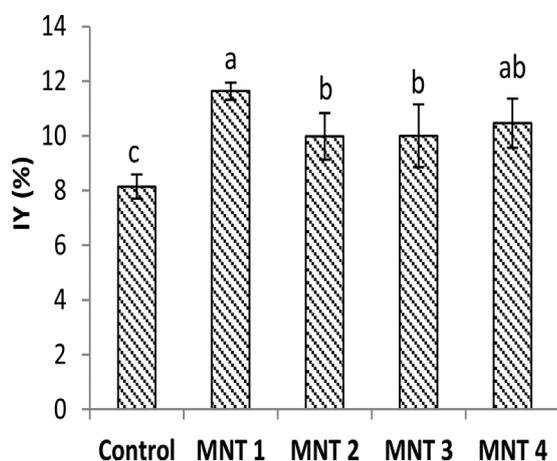


Figure 2. Effect of MNT physicochemical characteristics on the process industrial yield (IY) considering the four MNT doses together. Different letters means significant differences at p , 0.05.

Table 4 shows the percentage of the IY improvement obtained for each talc dose compared to the control. Results indicated that the oil yield improvement was not the same for the different MNTs. The most efficient talc, with an IY improvement of 26% at low dose (1%) was MNT1; while using the same dosage, MNT2 and MNT4 improved the IY only by 8.15 and 10.62%, respectively.

These differences observed in the IY as a response to MNT doses of each MNT tested may be explained by their different physicochemical properties (Table 2) which vary with their original deposit and preparation process. These results agree with those found by others authors [5, 22, 23] reporting as MNT physicochemical characteristics such as the particle size (D 50) and the carbonate rate play an important role in breaking oil-in-water emulsions and then improving the oil extraction process.

Lower D 50 and thus, greater active surface area allow a better adsorption of the droplets of oil retained in the cell walls, forming larger drops, thereby facilitating the extraction of this oil. Higher carbonate rate allows the enhancement of the breakdown of oil-in-water emulsions by releasing oil droplets that merge into larger drops as described by Alba et al. [23] and Sadkaoui et al. [5].

When IY is represented as function of carbonate content (Fig. 3A) can be observed that IY increased as the carbonate rate in the talc was higher. Furthermore, the effect of carbonate rate depended on the talc dose being much more pronounced at lower doses. The highest IY value was obtained using the higher MNT concentration (2 and 3%) of MNT with the highest carbonate rate (4%) while the lowest value was achieved using the smallest concentration (1%) of MNT with the lowest carbonate rate (0.5%). Therefore, MNTs with lower carbonate rate need higher doses to achieve IY similar to those obtained from low carbonate rate MNT.

Considering the particle size (Fig. 3B), higher D 50 seems to have a negative effect on the IY although it depended on the talc dose. At 1%, the extraction efficiency showed highest differences for smallest particle size. For higher doses, differences between MNT with higher carbonate rate and the others was lesser. This result suggests that higher doses are recommended to improve the extraction efficacy when MNT with high D 50 are used. In general, IY was greater when MNT with small particle size and high carbonate rate are used. This results confirms those published by Alba et al. [23], Sánchez et al. [22]; and Sadkaoui et al. [5] reporting that MNT containing high carbonate content and low D 50 shows a better capacity to break oil-in-water emulsions and gives higher oil yields.

According to Öksüz et al. [30], the surface hydrophobicity of the MNT increases as particle size is smaller due to an increase in the value of contact angles. Based on this finding, it can be deduced that MNT 1 had the highest hydrophobic surface area. Likewise, Casanova et al. [31] found that a decrease in particle size produces an increase in the surface

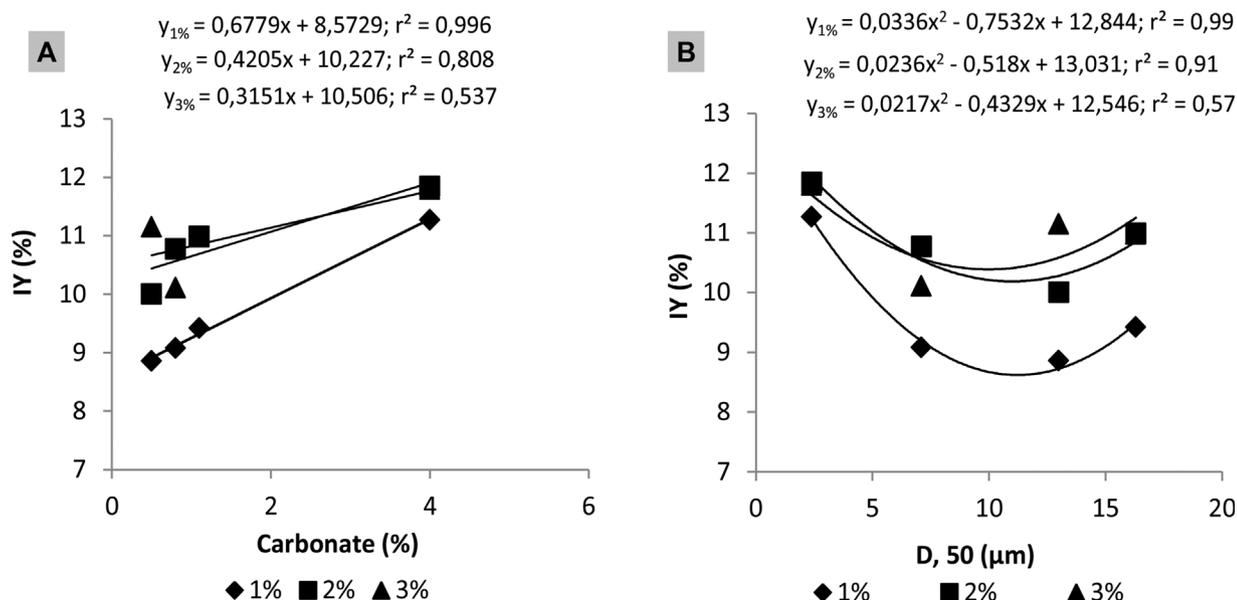


Figure 3. Correlation between the process industrial yield (IY) and MNT physicochemical characteristics applied at different doses. (A) Carbonate rate; (B) particle size.

area (S_{BET}) of the adsorbed oil. Therefore, IY may have a direct relationship with the S_{BET} of the talc. Because the effectiveness of the MNT depended not only on its surface area but also on its carbonate fraction (X_{Carb}) (as observed in this work) it is possible to bring these two terms together as S_{BET}/X_{Carb} in order to study its influence on the IY. Considering the four MNTs together, Fig. 4 shows as the increase of S_{BET}/X_{Carb} induced a lineal decrease of the IY,

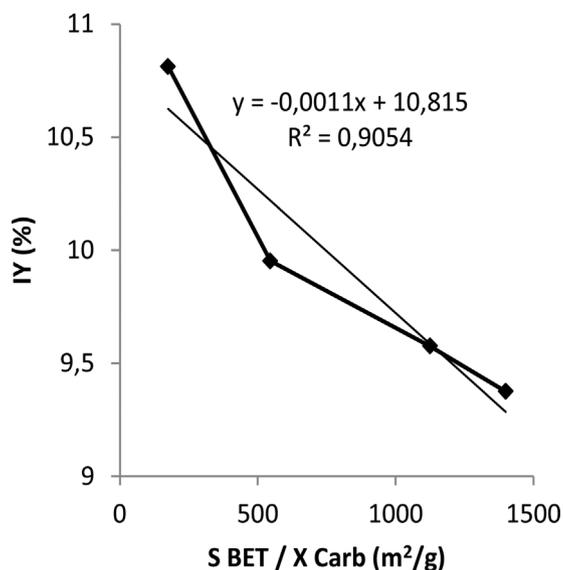


Figure 4. The process industrial yield (IY) as a function of the specific surface area/weight carbonate fraction (m^2/g).

then a close relationship between the IY and the S_{BET}/X_{Carb} was obtained ($p \leq 0,01$) with a r^2 value of 90%.

4 Conclusions

The use of MNT improves the IY. In general, the use of MNT doses ranging between 1 and 2% gave rise to a lineal increase of the IY whereas higher doses did not improve the oil extractability. The effectiveness of MNT to increase the IY depended not only on the dose but also on its physicochemical characteristics. The most efficient MNT was characterized by the highest carbonate rate and the lowest particle size achieving the highest improvement of the IY and oil loss for a dose of 2%. Besides, it was found that low carbonate levels or high particle sizes could be compensated with higher doses of talc. Therefore, MNT doses should be adapted to MNT physical and chemical characteristics in order to optimize the oil yield. These results can be easily scaled for its application at industrial scale since Abencor system has been considered as a useful method for olive oil extraction studies.

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