



Article Use of Natural Microtalcs during the Virgin Olive Oil Production Process to Increase Its Content in Antioxidant Compounds

Sebastián Sánchez ^{1,2,*}, Inmaculada Olivares ^{1,2}, Juan G. Puentes ^{1,2}, Rafael Órpez ^{1,2}, M. Dolores La Rubia ^{1,2}, Rafael Pacheco ^{1,2} and Juan F. García Martín ^{1,3,*}

- ¹ University Institute of Research on Olive Groves and Olive Oils, GEOLIT Science and Technology Park, University of Jaen, 23620 Mengibar, Spain; momerino@ujaen.es (I.O.); jpuentes@ujaen.es (J.G.P.); rorpez@ujaen.es (R.Ó.); mdrubia@ujaen.es (M.D.L.R.); rpacheco@ujaen.es (R.P.)
- ² Department of Chemical, Environmental and Materials Engineering, University of Jaen, 23071 Jaen, Spain
- ³ Departamento de Ingeniería Química, Facultad de Química, Universidad de Sevilla, 41012 Seville, Spain
 - * Correspondence: ssanchez@ujaen.es (S.S.); jfgarmar@us.es (J.F.G.M.)

Abstract: During the olive oil production process, certain olive varieties, such as 'Hojiblanca' and 'Picual', create pastes from which it is difficult to separate the oil, resulting in low extraction yields. To improve oil extraction, one alternative is the addition of natural microtalcs (NMT). In the present study, a NMT of great purity (CaCO₃ concentration less than 6 wt.%) and small average particle size ($\phi \leq 2.1 \mu$ m) was added in the malaxation stage on an industrial scale at two olive mills. In one of them and using 'Hojiblanca' olives, the performance of the high-purity NMT was compared with that of a traditional NMT, while in the other, the effect of its dosage in the quality of 'Picual' oils was assessed. The performance of the high-purity NMT was evaluated in terms of industrial oil yield, extractability index, quality parameters and oxidative stability of the resulting oils. The addition of the high-purity NMT not only increased the extraction yields but also improved the quality of the virgin olive oils, especially in relation to antioxidant compounds (tocopherols and phenolic compounds). Increases of 10.4% in phenolic compounds and of 21.5% in the tocopherols were found, thus enhancing the oxidative stability of the oils.

Keywords: extractability index; industrial oil yield; natural microtalc; oxidative stability; phenolic compounds; tocopherols; virgin olive oils

1. Introduction

Virgin olive oils are defined by the International Olive Council as "the oils obtained from the fruit of the olive tree (*Olea europaea* L.) solely by mechanical or other physical means under conditions, particularly thermal conditions, that do not lead to alterations in the oil, and which have not undergone any treatment other than washing, decantation, centrifugation, and filtration" [1].

In certain olive growing areas, there are serious difficulties in the olive oil production process with certain varieties of olives. In these cases, after the grinding stage, pastes are produced from which it is difficult to separate the fat matter (called "strong or difficult pastes"). At the macroscopic level, retention and occlusion of the liquid phases in the pastes, oil-in-water emulsions, and very fine solids in the form of a slurry are observed, resulting in lower extraction yields at the industrial scale.

An increase in the temperature used in the malaxation step usually improves the oil extraction yield because of the reduction of the oily phase viscosity. However, an excess of temperature during malaxation can jeopardize the nutritional and health values of extra virgin olive oil (EVOO), leading to undesirable effects, such as accelerating the oxidative process and the loss of volatile compounds responsible for the aroma of the oil [2].



Citation: Sánchez, S.; Olivares, I.; Puentes, J.G.; Órpez, R.; La Rubia, M.D.; Pacheco, R.; García Martín, J.F. Use of Natural Microtalcs during the Virgin Olive Oil Production Process to Increase Its Content in Antioxidant Compounds. *Processes* **2022**, *10*, 950. https://doi.org/10.3390/pr10050950

Academic Editor: Carla Silva

Received: 2 April 2022 Accepted: 6 May 2022 Published: 10 May 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). In order to improve the management of these difficult pastes in the production of olive oils without increasing the temperature and to comply with the definition of virgin olive oil, one of the widely accepted alternatives is the addition of a technological adjuvant to these pastes during the malaxation step. Among these adjuvants, the most used, proposed by the International Olive Council and accepted by the European Union, are natural microtalcs (NMT) [3,4].

The use of natural microtalcs (hydrated magnesium silicate) has been authorized by European regulation since 1986, solely for food purposes, provided that no changes in the physical and organoleptic properties of olive oil are produced. The current European Union Regulation allows a maximum concentration of 6 wt.% of calcium carbonate in NMT.

Initially and for three decades, the aim of adding microtalcs was only to increase the extraction yields without affecting the natural properties of the pastes or modifying the physical–chemical and sensory characteristics of the virgin oils obtained [5–7], keeping intact the nutritional quality of the oils obtained.

The addition of microtalcs promotes the breakdown of oil-in-water emulsions, thus facilitating the extraction of oil from mesocarp cells, as the oil does not remain in the pomace and consequently the oil yield increases [2,8,9]. This breakage of oil-in-water emulsions can be explained by the fact that the use of microtalcs during the malaxation step greatly lessens the action of pectic substrates and proteins, which are the main factors responsible for the formation of oil-in-water emulsions during the malaxation step [8]. Notwithstanding, excessive talc addition leads to a negative effect (taste loss) [8].

The main objective of the present work was to increase the industrial oil yield and extractability index and to improve the physicochemical composition of olive oils for olive varieties prone to forming difficult pastes by the addition of a new, commercially available, high-purity natural microtalc. A comparative study on the use of this high-purity NMT with a small average particle size ($\phi \leq 2.1 \,\mu$ m) in the industrial-scale production of virgin olive oils was carried out. Two varieties were assayed, namely 'Hojiblanca' and 'Picual', in which pastes are difficult to separate the fat matter (the olive oil). With the former, the performance of the high-purity NMT in the malaxation stage was compared with that of a traditional NMT. With the later, the efficiency of the process with the addition or without the addition of the high-purity NMT was assessed. The influence of the addition of the high-purity of the only on extraction yields, but also on the improving in the quality of virgin olive oils produced, especially in relation to antioxidant compounds (tocopherols and phenolic compounds) and thus on the oxidative stability of the olive oils.

2. Materials and Methods

2.1. Raw Materials

Olives from the variety 'Hojiblanca' were supplied by the olive oil mill 'Oleoalgaidas S.C.S.' (Villanueva de Algaidas, Malaga, Spain) during the 2011/2012 harvesting, while 'Picual' olives were supplied by the olive oil mill 'Aceites San Antonio S.L.U.' (Escañuela, Jaen, Spain) during the 2016/2017 harvesting.

Two natural microtalcs were used, both authorized for food use. The first NMT was the FC8KN natural microtalc, supplied by Mondo Minerals BV (Amsterdam, The Netherlands), which was used for the production of virgin olive oils in both harvestings. This NMT is characterized by its high purity (97 wt.%, calcium carbonate concentration less than 6 wt.%) and small particle size (with a D98% top cut of 8.6 μ m, and an average particle size for D50% of 2.1 μ m). Its BET specific surface area (ISO 4652) is 7 m²/g, as indicated by the supplier. This means that it can be added in a lower percentage to olive pastes compared to traditional microtalcs. For comparison, a traditional NMT (17.5 μ m, D90% < 40.0 μ m, calcium carbonate concentration not specified) was also used in the production of virgin olive oils with 'Hojiblanca' olives during the 2011/12 harvesting.

2.2. Oil Olive Production at Industrial Scale

The sampling was carried out at different times in the olive ripening process during the 2011/12 and 2016/17 harvest seasons. This research was carried out on an industrial scale in the production lines of two olive oil mills that use the continuous centrifugation process with two-outlet decanters. One olive oil mill was 'Oleoalgaidas S.C.A.' (Villanueva de Algaidas, Malaga, Spain) from 'Grupo DCOOP' and the other was 'Aceites San Antonio S.L.U.' (Escañuela, Jaen, Spain).

Three- and four-body malaxers were used in the malaxation step process, 90 min being the malaxation time and 5000 kg h⁻¹ the mass flow rate of the paste for the 'Oleoalgaidas S.C.A.' oil mill (Figure 1) and 60 min the malaxation time and 3000 kg h⁻¹ the mass flow rate of the paste for the 'Aceites San Antonio S.L.U.' oil mill (Figure 2). The natural microtalcs were continuously added through dosage equipment during the malaxation step, before the centrifugation process of the olive pastes, at a percentage of 0.3 wt.% in relation to the mass flow rate of the paste in the production line. The NMT dispenser was placed at the beginning of the last body of the malaxer, because it was found in preliminary assays that this position was the one that led to a greater increase in industrial yields (data not shown). Two samples of each produced oil were collected at the outlet of the horizontal centrifuge in a time interval of less than 1 h.



Figure 1. Malaxers and decanters in two different lines of the olive oil extraction process in the 'Oleoalgaidas S.C.A.' olive mill.



Figure 2. Malaxer and NMT dosage equipment at the 'Aceites San Antonio S.L.U.' olive mill.

2.3. Industrial Oil Yield and Extractability Index

About 750 g olives from each sample batch were taken to the laboratory and ground in an MM-100 hammer mill of an Abencor ('MC2 Ingeniería y Sistemas S.L.', Seville, Spain) mini-plant system for olive oil production, as described elsewhere [10], which is widely accepted to obtain the actual values of oil yield at the industrial scale. The extractability index is defined as the ratio between the industrial yield and the content of total fat matter on a wet basis, and is usually expressed as a percentage [11]. The total fat matter on a wet basis was determined by extraction in Soxhlet equipment with technical hexane.

2.4. Analytical Methods

The olive oils obtained were characterized according to the following quality parameters: free acidity, peroxide value, and ultraviolet absorption (K₂₇₀, K₂₃₂ and Δ K). These parameters were determined following the analytical methods described in the CEE/2568/91, CEE/1429/92 regulations and subsequent modifications of the European Commission [12,13]. A brief description of the procedures can be found elsewhere [14]. The free acidity, expressed as percentage of oleic acid, was determined by placing the olive oil in an ethanol/ethyl ether solution (1:1 v/v) along with a few drops of phenolphthalein and then neutralized with 0.1 M KOH.

Regarding the peroxide value, expressed in $mEqO_2$ per kg of oil, it was determined by letting a mixture of oil and acetic acid chloroform react in the dark with a solution of potassium iodide; then, free iodine was titrated with a sodium thiosulfate solution.

The ultraviolet absorption parameters, K_{232} , K_{270} , and ΔK (absorption of a 1% solution of oil in cyclohexane at 232 and 270 nm), were measured in a UV-VIS spectrophotometer Spectronic Helios γ (Thermo Fisher Scientific, Waltham, MA, USA).

Similarly, the content of total phenolic compounds was determined as well as the oxidative stability of the oils produced. Regarding the evaluation of oxidative stability,

the oxidation induction time was measured by Rancimat equipment, Mod. 743 (Metrohm Hispania, Madrid, Spain). Briefly, 3 g of oil were weighed and, after heating to 98 °C, an airflow was bubbled through it with a volumetric flow rate of 10 dm³·h⁻¹. The results obtained referring to the oxidative stability were expressed as induction time in hours [15]. On the other hand, the content of total phenolic compounds was determined by extraction with methanol:water (60:40 v/v) solution and measurement of the absorbance of the complex formed between phenolic compounds and the Folin-Ciocalteu reagent at λ = 725 nm in a UV-VIS spectrophotometer Spectronic Helios γ (Thermo Fisher Scientific, Waltham, MA, USA), as described elsewhere [16].

The contents of α -, β -, γ -, δ - and total tocopherols were determined by high-performance liquid chromatography following a standardized method [17]. For this purpose, a Prominence modular HPLC system (Shimadzu Corporation, Kyoto, Japan), equipped with a fluorescence detector and a LiChrospher 5 µm Sil 60A analytical column (Phenomenex, Torrance, CA, USA) were used. Propan-2-ol in n-hexane (0.5:99.5 v/v) was used as mobile phase with a flow of 0.5 cm⁻³ min⁻¹ and a temperature of 24 °C.

3. Results and Discussion

3.1. Industrial Oil Yield

Higher industrial oil yields were achieved with the 'Picual' variety than with 'Hojiblanca' variety (data not shown). Initially, this fact is logical, since 'Picual' olives have higher fat matter content. Based on the experimental results, an increase in the industrial oil yield in oil was observed when the FC8KN natural microtalc was added during the malaxation step at different times of the elaboration process (Figure 3).



Figure 3. Industrial oil yield for the 'Picual' variety at different times during the 2016/2017 harvesting without (**■**) and with (**■**) FC8KN natural microtalc addition during the malaxation step.

In general, the addition of the FC8KN natural microtalc improved the industrial oil yield in all cases compared to the control without the addition of NMT. The average increase obtained for the samples shown in Figure 3 was 25%, which is similar to the increases found by other authors on the use of NMT but with higher dosages [18,19] and other olive varieties [19–21]. Of note is the increase in industrial oil yield obtained for the sample from 26 January 2017. The extraction process without NMT rendered an industrial oil yield of 11.34 wt.%, while it rose to 17.65 wt.% when using the FC8KN natural microtalc, which represents an increase of 56%. This increase is much higher than the highest increase in industrial oil yield (32% using 1 wt.% NMT and 'Hojiblanca' variety) that can be found in the literature [18]. Since the NMT dosage used in this work (0.3 wt.%) is even lower than those recommended in the available literature (0.5–2.0 wt.%) [22,23], the high industrial oil yields obtained can be attributed to the physicochemical characteristics of the FC8KN natural microtalc [18,23,24].

Physicochemical characteristics such as particle size (D50) and microtalc purity play an important role in breaking oil-in-water emulsions and improve the oil extraction process. The ability of microtalc to increase oil yields could be explained by its physical action, since it adsorbs the oil droplets retained in the cell walls, forming larger droplets and therefore facilitating oil extraction [25]. In olive paste, oil and water can be trapped in agglomerating microgels. Additionally, both paste and microtalc have lipophilic properties, so they tend to retain oil. The microtalc facilitates the release of the oil in an extractable form, breaking the reticular structure formed by the links among microgels [4,26,27].

Regarding the degree of depletion of the pomaces produced and with varieties very prone to forming difficult pastes ('Hojiblanca' and 'Picual'), values very similar to those reached at the laboratory level were obtained at the industrial level in both oil mills (2011/12 and 2016/17 harvestings, respectively). These results agree with those found by other authors using the 'Hojiblanca' variety in a process with a two-outlet decanter [22]. Additionally, it should be noted that the difference in consumption between both microtalcs was significant at each of the sampling times assayed (Figure 4). Thus, a reduction of more than 40% in used NMT was achieved in the experience of 12 January 2012 when using the FC8KN natural microtalc.



Figure 4. Mass flow of traditional (**I**) and FC8KN (**I**) natural microtalcs in processes with olives of the 'Hojiblanca' variety.

Traditional microtalcs, large in size, are usually added at the beginning of the malaxation stage together with the paste. However, the FC8KN microtalc was added at the beginning of the last body of the malaxer (corresponding to the last third of the malaxation total time), reducing the contact time between NMT and pastes and increasing oil yields and extractability indexes. Although the microtalc is a natural product, it is advisable to reduce at minimum the contact time between the olive paste and the technological adjuvant, since the microtalc is not a component of the olives.

In relation to the extractability index, extractability indexes over 80% were achieved with FC8KN microtalc for both varieties. To be specific, an extractability index value of 88.9% was obtained from olives of the 'Picual' variety with a maturity index of 5.62, an average fruit weight of 2.47 g, and a pulp/stone ratio of 2.17.

In general, it was demonstrated that when an olive paste has a water percentage between 48 and 51 wt.%, the addition of a NMT leads to an increase in the extractability index (data not shown), mainly when the FC8KN microtalc was used.

A visual aspect observed in the olive oils at the exit of the decanter was that they were cleaner (free of solid particles) and had higher pigmentation (greener). A similar fact occurred at the oultlet of the horizontal centrifuge. This fact could be attributed to a higher content of chlorophyll pigments and a lower content of water microdroplets and solid microparticles.

3.2. Effect of the Additon of FC8KN Natural Microtalc on Olive Oil Quality Parameters

From the results obtained in the characterization of the olive oils of both varieties ('Picual' and 'Hojiblanca') according to the olive oil quality parameters, namely acidity, peroxide index, and UV absorption, it is observed that all of them comply with the standards of the European Commission for edible oils [1]. In fact, 'Hojiblanca' oils produced before December 30 are within the normal ranges for EVOO, while oils obtained after December 30 are considered virgin olive oils (Table 1). As for 'Picual' oils, all are in accordance with the limits to be labelled as EVOO (Table 2).

Table 1. Quality parameters of olive oils obtained by adding FC8KN natural microtalc to olive pastes of the 'Hojiblanca' variety.

Haversting Date	Acidity (%)	Peroxide Value (mEq O ₂ /kg)	K ₂₃₂	K ₂₇₀	ΔΚ
22 Dec, 2011	0.20 ± 0.00	16.94 ± 2.96	1.53 ± 0.01	0.13 ± 0.01	0.01 ± 0.01
23 Dec, 2011	0.24 ± 0.02	15.08 ± 0.77	1.51 ± 0.12	0.12 ± 0.02	0.00 ± 0.00
30 Dec, 2011	0.23 ± 0.04	16.61 ± 0.01	1.48 ± 0.05	0.11 ± 0.00	0.00 ± 0.00
4 Jan, 2012	0.20 ± 0.03	22.84 ± 1.61	1.58 ± 0.06	0.10 ± 0.02	0.00 ± 0.00
12 Jan, 2012	0.20 ± 0.00	17.53 ± 0.00	1.62 ± 0.00	0.12 ± 0.00	0.00 ± 0.00
20 Jan, 2012	0.21 ± 0.02	20.52 ± 1.75	1.55 ± 0.04	0.12 ± 0.02	0.00 ± 0.00
Limits fixed by the IOC [1]	≤ 0.80	\leq 20.0	≤2.50	≤0.22	≤ 0.01

Table 2. Quality parameters of olive oils obtained by adding FC8KN natural microtalc to olive pastes of the 'Picual' variety.

Haversting Date	Acidity (%)	Peroxide Value (mEq O ₂ /kg)	K ₂₃₂	K ₂₇₀	ΔΚ
10 Jan, 2017	0.17 ± 0.00	2.93 ± 0.04	1.56 ± 0.04	0.18 ± 0.02	0.00 ± 0.00
18 Jan, 2017	0.17 ± 0.01	3.65 ± 0.06	1.64 ± 0.01	0.13 ± 0.00	0.00 ± 0.00
23 Jan, 2017	0.19 ± 0.02	4.77 ± 0.29	1.74 ± 0.01	0.15 ± 0.00	0.00 ± 0.00
26 Jan, 2017	0.17 ± 0.00	2.67 ± 0.28	1.73 ± 0.01	0.16 ± 0.00	0.00 ± 0.00
1 Feb, 2017	0.18 ± 0.01	3.83 ± 0.18	1.55 ± 0.03	0.12 ± 0.00	0.00 ± 0.00
23 Feb, 2017	0.28 ± 0.00	4.76 ± 0.23	1.59 ± 0.03	0.11 ± 0.01	0.00 ± 0.00
Limits fixed by the IOC [1]	≤ 0.80	\leq 20.0	≤2.50	≤0.22	≤0.01

On the other hand, it should be noted that the addition of the FC8KN natural microtalc has resulted, with respect to the characterization of the olive oils obtained, in lower values in these quality parameters when compared with those obtained without the addition of this natural adjuvant. On the other hand, if these quality parameters are compared with the addition of a different microtalcs (traditional NMT vs. FC8KN NMT), it was observed that the values of acidity, peroxide value, and UV absorption were very similar for the 11/12 harvesting with 'Hojiblanca' [28]. However, the peroxide value was slightly lower in oils produced using the FC8KN natural microtalc, results that can be found in that previous study. These results confirm the findings of other authors using different olive varieties, who have reported that microtalc addition protects the oil from oxidation [19,21,29,30].

From the results obtained in the determination of phenolic compounds, it is highlighted that there is a higher concentration in the olive oils obtained in production processes with the addition of NMT. This effect is clearly reflected in the 2016/2017 campaign and for the 'Picual' variety (Figure 5). In this research, increases of 10.4% in the fraction of total phenolic compounds were achieved when the FC8KN natural microtalc was added in the malaxation stage of the olive oil production process.



Figure 5. Concentration of total phenolic compounds in oils of the 'Picual' variety at different times of the 2016/2017 harvesting, produced without (**■**) and with the addition (**■**) of the FC8KN natural microtalc during the malaxation stage in the oil extraction process.

3.3. Effect of the Additon of FC8KN Natural Microtalc on Olive Oil Tocopherol Content

In this research, the total tocopherol content of the olive oils obtained from 'Picual' variety (2016/2017 harvesting) was close to or greater than 300 mg/kg oil when using the FC8KN NMT, reaching 443.4 mg/kg oil in one of the periods (Figure 6). These values are in agreement with the work of Cayuela and García [31], who reported a range of 55.2–466.4 mg/kg oil for the concentration of total tocopherols.



Figure 6. Concentration of total tocopherols in oils of the 'Picual' variety at different times of the 2016/17 harvesting, produced without (•) and with addition (•) of the FC8KN natural microtalc during the malaxation stage.

The effect of microtalc addition on the tocopherol content of olive oils has not been extensively studied to date, and most studies found a significant decrease in the tocopherol content when adding microtalc in the malaxation stage [19,21]. By contrast, increases of 21.5% in the fraction of total tocopherols and of 22.1 and 25.6% in α - and β -tocopherol, respectively, were obtained when the FC8KN NMT was used in the malaxation stage of the extraction process of the 'Picual' variety (Table 3) when comparing with olive oils from the same olive batches produced without the addition of NMT. This result could be attributed to the lower particle size and higher purity of the FC8KN microtalc.

Table 3. Average increase in the concentration of tocopherols in olive oils from the 'Picual' variety, produced at different times of the 2016/2017 harvesting, when adding the FC8KN natural microtalc during the malaxation step.

Harvesting Date	%Δ α-Tocopherol	%Δ β-Tocopherol	%Δ γ-Tocopherol	%Δ Total Tocopherols
10 Jan, 2017	9.5	10.3	-12.9	7.8
13 Jan, 2017	5.3	5.5	9.8	5.5
18 Jan, 2017	4.6	25.6	10.2	6.0
23 Jan, 2017	10.0	7.8	11.2	10.0
26 Jan, 2017	13.1	10.5	15.4	13.1
1 Feb, 2017	22.1	25.3	7.1	21.5
7 Feb, 2017	8.6	6.6	16.9	9.0
9 Feb, 2017	4.1	1.0	1.6	3.7
16 Feb, 2017	17.6	9.0	13.6	17.0
21 Feb, 2017	16.9	6.4	5.8	15.9
23 Feb, 2017	4.7	5.2	15.6	5.1

Tocopherols have an important activity as natural antioxidant agents and confer stability to the oils that contain them. Consequently, they are important constituents in virgin olive oils, since they give it stability and they have a beneficial biological role as antioxidants. The α -tocopherol (90–95% of the total tocopherol content) is the main homologue of the forms of vitamin E that exerts an important antioxidant action, since it reacts with the peroxide radicals of the fatty acids, which are the primary products of fat auto-oxidation, thus stopping spoilage in the early stages. In general, the contents of the β and γ forms are below 10% [32]. δ -tocopherol is usually present in such low concentrations that some authors even state that this form is not present in olive oils [33,34]. In this sense, in the trials carried out in this work, δ -tocopherol has not been detected in any of the sampling periods.

3.4. Effect of the Additon of Natural Microtalc on Olive Oil Oxidative Stability

Regarding the evaluation of oxidative stability, the Rancimat method is an accelerated stability test that provides very useful information about the resistance of the oil to the oxidation process. In general, a higher stability can be observed in the oils produced with smaller particle size microtalc (Figure 7). When traditional NMT was used in the process, the average oxidative stability for the oils produced from the 'Hojiblanca' variety was 50.6 h, with a range between 21.1 and 83.1 h, while using the FC8KN natural microtalc the average oxidative stability in the oils produced was 67.7 h, with a range between 34 and 107.9 h. These last results are very close to those obtained by Beltrán et al. with the same variety ('Picual'), in a process at the mini-plant level (Abencor system), during three consecutive years of harvesting, and using a natural microtalc [35].



Figure 7. Oxidative stability of virgin olive oils of the 'Hojiblanca' variety produced using the FC8KN (■) and a traditional (■) NMT.

4. Conclusions

The FC8KN natural microtalc assayed in this work is characterized by its small particle size and extraordinary purity. These characteristics lead to a lower percentage added to the olive pastes compared to a traditional NMT; the FC8KN natural microtalc dosage (0.3 wt.%

in relation to the mass flow rate of the paste in the production line) was lower than those reported in the available literature (between 0.5 and 2.0 wt.%).

In the production of olive oils from both the 'Picual' and 'Hojiblanca' varieties, the industrial yield and extractability index increased when a natural microtalc was added during the malaxation stage. The olive oil yield increased by 25% on average when using the NMT with a smaller particle size (FC8KN). A maximum of 56% increasing in industrial oil yield was achieved with FC8KN when compared with the same process without the addition of NMT. This increase in industrial oil yield is much higher than those reported by other authors on the use of other microtalcs and at higher dosages.

Taking into account the quality parameters, the olive oils produced adding the FC8KN natural microtalc showed lower values (therefore higher quality) in these parameters when compared to those obtained without the addition of NMT. It is worth noting that improved pigmentation (higher concentration of chlorophylls) was visually observed for both the 'Picual' and 'Hojiblanca' varieties when adding FC8KN microtalc.

Finally, olive oils produced with the FC8KN natural microtalc showed a higher content of phenolic compounds and thus higher oxidative stability, both in the 'Picual' and 'Hojiblanca' varieties. To be specific, increases of up to 10.4% in total phenolic compounds, 21.5% in total tocopherols, and 22.1% in α -tocopherol were achieved when the FC8KN natural microtalc was added in the malaxation stage of the olive oil production process.

All in all, the FC8KN natural microtalc should be used in the olive oil industry to obtain extra virgin olive oils with high quality from olive varieties prone to forming difficult pastes.

Author Contributions: Conceptualization, S.S., R.P. and J.F.G.M.; methodology, S.S., M.D.L.R., R.Ó., I.O. and J.G.P.; software, validation, and formal analysis, J.F.G.M. and I.O.; investigation and data curation, R.Ó., M.D.L.R., I.O. and J.G.P.; resources, R.P., M.D.L.R., R.Ó. and I.O.; writing—original draft preparation, S.S., J.F.G.M. and I.O.; writing—review and editing, S.S., J.F.G.M. and M.D.L.R.; visualization, J.F.G.M., I.O. and J.G.P.; supervision: S.S., J.F.G.M. and I.O.; project administration, S.S. and R.P.; funding acquisition, S.S. All authors have read and agreed to the published version of the manuscript.

Funding: The authors acknowledge the industry Mondo Minerals BV (Barcelona, Spain) and the Andalusia Regional Government (Spain) for its financial support to our research group 'Bioprocesses TEP-138'.

Informed Consent Statement: Not applicable.

Acknowledgments: The authors are grateful to the industries Mondo Minerals BV (Barcelona, Spain), 'Aceites San Antonio S.L.U.' (Escañuela, Jaen, Spain), and 'Oleoalgaidas S.C.A.' (Villanueva de Algaidas, Malaga, Spain).

Conflicts of Interest: The authors declare no conflict of interest.

References

- IOC/T.15/NC No 3/ Rev.17/2021; Trade Standard Applying to Olive Oils and Olive Pomace Oils. International Olive Council: Madrid, Spain, 2021; pp. 1–17.
- Caponio, F.; Squeo, G.; Difonzo, G.; Pasqualone, A.; Summo, C.; Paradiso, V.M. Has the use of talc an effect on yield and extra virgin olive oil quality? J. Sci. Food Agric. 2016, 96, 3292–3299. [CrossRef] [PubMed]
- Martínez, F.; Moyano, M.J.; Alba, J.; Ruiz, M.A.; Hidalgo, F.; Heredia, F.J. Método rápido de obtención de aceite de oliva virgen para determinación de acidez. Grasas Aceites 1999, 50, 122–126. [CrossRef]
- Sánchez, S.; Rubia, M.D.; Sánchez, A.; Pereira, M.G. Evolución histórica de la utilización de coadyuvantes tecnológicos en la producción de aceite de oliva. In Proceedings of the 'I Congreso de Cultura del Olivo', Jaén, Spain, 27–29 October 2005; pp. 623–633.
- 5. Di Giovacchino, L. L'impiego del talco micronizzato nell' estrazione dell' olio dalle olive con il sistema continuo. *Riv. Ital. Delle Sostanze Grasse* **1988**, 65, 623–628.
- 6. Alba, J.; Muñoz, E.; Martínez, J.M. Obtention of olive oil use of additives facilitating its extraction. Alimentaria 1982, 138, 25–55.
- Alba, J. Obtención del aceite oliva. Empleo de productos que facilitan su extracción. In Proceedings of the XVIII Reunión Plenaria de la Asamblea del Instituto de la Grasa y sus Derivados, Seville, Spain, 15–17 October 1982.
- Sadkaoui, A.; Jiménez, A.; Pacheco, R.; Beltrán, G. Micronized natural talc affects the proteins and pectic cell wall polysaccharides during "Hojiblanca" virgin olive oil extraction. *Eur. J. Lipid Sci. Technol.* 2017, 119, 1600039. [CrossRef]

- Di Giovacchino, L. I sottoprodotti della lavorazione delle olive in oleificio. In *Tecnologie di Lavorazione Delle Olive in Frantoio*; Di Giovacchino, L., Ed.; Tecnica Alimentare: Milano, Italy, 2010; pp. 243–258. ISBN 978-88-481-2554-3.
- 10. Plasquy, E.; García Martos, J.M.; Florido Fernández, M.D.C.; Sola-Guirado, R.R.; García Martín, J.F. Adjustment of olive fruit temperature before grinding for olive oil extraction. Experimental study and pilot plant trials. *Processes* **2021**, *9*, 586. [CrossRef]
- 11. Beltrán, G.; Uceda, M.; Jiménez, A.; Aguilera, M.P. Olive oil extractability index as a parameter for olive cultivar characterisation. *J. Sci. Food Agric.* 2003, *83*, 503–506. [CrossRef]
- 12. Commission Regulation (EEC) No 2568/91 on the characteristics of olive oil and olive-residue oil and on the relevant methods of analysis. *Off. J. Eur. Communities* **1991**, *L248*, 1–15.
- Commission Implementing Regulation (EU) No 1348/2013 amending Regulation (EEC) No 2568/91. Off. J. Eur. Union 2013, L338, 31–67.
- 14. García Martín, J.F. Potential of near-infrared spectroscopy for the determination of olive oil quality. *Sensors* **2022**, *22*, 2831. [CrossRef]
- 15. Gutierrez Rosales, F. Determination of oxidative stability in virgin olive oils: Comparison between active oxygen method and Rancimat method. *Grasas Aceites* **1989**, *40*, 1–5.
- 16. Vázquez-Roncero, A.; Janer del Valle, C.; Janer del Valle, M.L. Determinación de los polifenoles totales del aceite de oliva. *Grasas Aceites* **1973**, 24, 350–357.
- Pocklington, W.D.; Dieffenbacher, A. Determination of tocopherols and tocotrienols in vegetable oils and fats by high performance liquid chromatography: Results of a collaborative study and the standardised method. *Pure Appl. Chem.* 1988, 60, 877–892. [CrossRef]
- Sadkaoui, A.; Jiménez, A.; Aguilera, M.P.; Pacheco, R.; Beltrán, G. Virgin olive oil yield as affected by physicochemical talc properties and dosage. *Eur. J. Lipid Sci. Technol.* 2017, 119, 1600112. [CrossRef]
- 19. Carrapiso, A.I.; García, A.; Petrón, M.J.; Martín, L. Effect of talc and water addition on olive oil quality and antioxidants. *Eur. J. Lipid Sci. Technol.* 2013, *115*, 583–588. [CrossRef]
- Aguilera, M.P.; Beltran, G.; Sanchez-Villasclaras, S.; Uceda, M.; Jimenez, A. Kneading olive paste from unripe "Picual" fruits: I. Effect on oil process yield. J. Food Eng. 2010, 97, 533–538. [CrossRef]
- 21. Caponio, F.; Monteleone, J.I.; Martellini, G.; Summo, C.; Paradiso, V.M.; Pasqualone, A. Effect of talc addition on the extraction yield and quality of extra virgin olive oils from Coratina cultivar after production and during storage. *J. Oleo Sci.* **2014**, *63*, 1125–1132. [CrossRef]
- Cert, A.; Alba, J.; León-Camacho, M.; Moreda, W.; Pérez-Camino, M.C. Effects of talc addition and operating mode on the quality and oxidative stability of virgin olive oils obtained by centrifugation. J. Agric. Food Chem. 1996, 44, 3930–3934. [CrossRef]
- Sánchez, S.; Pacheco, R.; La Rubia, M.D.; Sánchez, A.; Pereira, G. Aplicación de distintos talcos naturales, como coadyuvantes tecnológicos, en los procesos de extracción de aceites de oliva. In Proceedings of the XII Simposium Científico-Técnico Expoliva, Jaen, Spain, 12–13 May 2005; p. TEC-65.
- Sadkaoui, A.; Jiménez, A.; Pacheco, R.; Beltrán, G. Micronized natural talc with a low particle size and a high carbonate rate is more effective at breaking down oil-in-water emulsion. *Eur. J. Lipid Sci. Technol.* 2016, 118, 545–552. [CrossRef]
- Clodoveo, M.L.; Durante, V.; La Notte, D.; Punzi, R.; Gambacorta, G. Ultrasound-assisted extraction of virgin olive oil to improve the process efficiency. *Eur. J. Lipid Sci. Technol.* 2013, 115, 1062–1069. [CrossRef]
- 26. Martínez Moreno, J.M. Fundamentos Físico-Químicos de la Técnica Oleícola; C.S.I.C.—Patronato Juan de la Cierva: Madrid, Spain, 1972.
- 27. Nushtaeva, A.V. Emulsion stabilization with talc microparticles. Colloid J. 2019, 81, 425–430. [CrossRef]
- Sánchez, S.; Landeta, J.M.; Olivares, I.; Relaño, P.L.; Pacheco, R.; La Rubia, M.D. Use of natural microtalcs in the elaboration of virgin olive oil of 'Hojiblanca' variety. In Proceedings of the XVI Simposium Científico-Técnico Expoliva, Jaen, Spain, 8–12 May 2013; p. IND-58.
- 29. Ben-David, E.; Kerem, Z.; Zipori, I.; Weissbein, S.; Basheer, L.; Bustan, A.; Dag, A. Optimization of the Abencor system to extract olive oil from irrigated orchards. *Eur. J. Lipid Sci. Technol.* **2010**, *112*, 1158–1165. [CrossRef]
- Cruz, S.; Yousfi, K.; Pérez, A.G.; Mariscal, C.; Garcia, J.M. Salt improves physical extraction of olive oil. *Eur. Food Res. Technol.* 2007, 225, 359–365. [CrossRef]
- Cayuela, J.A.; García, J.F. Sorting olive oil based on alpha-tocopherol and total tocopherol content using near-infra-red spectroscopy (NIRS) analysis. J. Food Eng. 2017, 202, 79–88. [CrossRef]
- Lozano Sánchez, J. Aceite de Oliva Como Alimento Funcional: Nuevas Perspectivas Analíticas y Tecnológicas. Ph.D. Thesis, University of Granada, Granada, Spain, 2013.
- 33. Dionisi, F.; Prodolliet, J.; Tagliaferri, E. Assessment of olive oil adulteration by reversed-phase high-performance liquid chromatography/amperometric detection of tocopherols and tocotrienols. *J. Am. Oil Chem. Soc.* **1995**, *72*, 1505–1511. [CrossRef]
- 34. Rovellini, P.; Azzolini, M.; Cortesi, N. Tocoferoli e tocotrienoli in oli e grassi vegetali mediante HPLC. *Riv. Ital. Delle Sostanze Grasse* **1997**, 74, 1–5.
- 35. Beltrán, G.; Aguilera, M.P.; Del Rio, C.; Sánchez, S.; Martínez, L. Influence of fruit ripening process on the natural antioxidant content of Hojiblanca virgin olive oils. *Food Chem.* **2005**, *89*, 207–215. [CrossRef]