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## Sensory and physicochemical characteristics of Uruguayan picual olive oil obtained from olives with different ripening indexes

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#### Abstract

The sensory and physicochemical characteristics of olive oil will be mainly influenced by the agronomic conditions of the crop and harvest. Therefore, the results cannot be extrapolated from one region to another. On the contrary, the ripening index (RI) is a factor that can influence the quality of the oils obtained. Picual is one of the four most important olive varieties from Uruguay. This work studied how ripening influences the sensory and physicochemical characteristics of the extra virgin olive oil extracted from the Picual variety olives harvested in Uruguay. A total of 19 samples of olive oils extracted in the Abencor System pilot plant were analyzed. They were obtained from green, medium, and ripened olives from two different harvests (2021 and 2022). All the studied pastes were considered "difficult" due to a high moisture content of over 55%. This was reflected in the low yields of extraction which did not exceed 12.9%. All the oils obtained were characterized as extra virgin from a sensory and physicochemical point of view. Their content of total phenols and oleic acid allows defining Uruguayan Picual oils as "high in total phenols" and "high in oleic acid," respectively. They were sensory characterized as oils with fruity medium, bitter medium, and pungent medium and green, tomato, banana, and dry fruit aromatic notes. When olives increased ripening, the fruity, bitter, and pungent of the oils and phenol and oleic contents and the oleic/linoleic ratio decreased, and the yield was not modified. This is what makes the Uruguayan olive oil of the Picual variety ideal to be harvested with low RIs.

Keywords: olive oil; Picual; sensory; polyphenols; fatty acid.

**Practical Application:** The results obtained in the present study can be considered of great importance to position the extra virgin olive oils obtained from the Uruguayan variety Picual, as "high in total phenols," "high in oleic acid" and with a sensory profile with médium fruitiness, medium bitterness, and medium pungency and with green, tomato, banana, and dry fruit aromatic notes. Moreover, it provides information on the evolution of sensory and physicochemical quality along the fruit ripening, in order to help producers choose the optimum moment for harvesting.

### **1 INTRODUCTION**

Olive oil (*Olea europaea L.*) is one of the most important products in the Mediterranean diet. It has unique characteristics of sensory and chemical attributes and health benefits. Due to the high contents of monounsaturated fatty acids and the presence of phenolic compounds, virgin olive oil (VOO) has valuable antioxidant and anticancerogenic properties and a protective activity against heart disease, osteoporosis, and cognitive impairment (Fernandes et al., 2020; Rodríguez-Morató et al., 2015; Visioli et al., 2018). Nowadays, dietetic care and healthy nutrition habits, such as consumption of extra virgin olive oil (EVOO), are increasing among consumers, especially antioxidant intake, due to its long- and short-term benefits (Franco et al., 2014).

Currently, consumption of EVOO is not limited to European or Mediterranean countries but is a demanded product worldwide (Ouni et al., 2016). The increasing demand of EVOO can be attributed not only to its healthy effects but also to its sensory attributes which make EVOO distinctive from other vegetable oils (Konuskan & Mungan, 2016).

The sensory and physicochemical characteristics of olive oil will be influenced by the agronomic conditions of the crop (type of crop, RI, agroclimatic conditions, and agronomic practices), the raw material, the harvest, fruit storage, and extraction technology (Sena-Moreno et al., 2018; Sicari, 2017). The RI is an essential factor to be considered to obtain a better quality of EVOO.

During ripening, different metabolic pathways take place, which leads to several changes in the composition that affect fatty acids and phenolic content, among other compounds (Bakhouche et al., 2015), and also affect the sensory characteristics of VOOs.

Different varieties can evolve diversely during ripening, and thus, the results should not be extrapolated from one variety to another (Deiana et al., 2019; Gougoulias et al., 2017). Although there are contradictions, there are several studies that

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focused on the evolution of such parameters along olive drupes of different varieties (Navajas-Porras et al., 2020).

In Uruguay, currently, there are 162 productive exploitations of olives in 5.916 hectares mainly concentrated in the eastern area of the country with a production of around 1.900 tons of olive oil (Gorga & Ackermann, 2021; MGAP, 2020). Four varieties cover 90% of the cultivated area. Arbequina has the greatest area with 47% of the surface, followed by Coratina with 21% and Picual together with Frantoio with 11% each (Conde-Innamorato et al., 2019). According to the studies held by the Instituto Nacional de Investigación Agropecuaria (INIA), Picual is one of the olive varieties with a better productive yield in the country (Arias-Sibillote et al., 2021). It is considered the most important olive variety of Andalucía and cultivated in Spain, the European Union (UE), and worldwide (Aguilera et al., 2010). Moreover, the Picual variety is of great importance for the olive oil industry, due to the high yield for obtaining oil (García & Mancha, 1992). Despite the importance of Picual variety, there is a lack of data on its behavior.

Montaño et al. (2016) reported that the Picual variety has a higher content of polyphenols than other varieties such as the Arbequina variety.

The antecedents of olive oil extracted from the Picual olives have been exclusively done with olives cultivated in Spain, which reveals how kneading conditions affect the yield in oil extraction when fruits are early harvested (Aguilera et al., 2010), the influence of the ripening process of the fruit in the sensory quality (Jiménez Herrera et al., 2012), the evolution, during ripening, of parameters of physicochemical quality such as fatty acid composition, antioxidant capacity, and total phenolic content (Navajas-Porras et al., 2020), and its sensory and physicochemical characteristics (Diarte et al., 2021).

In this context, this study aims to know how ripening influences the sensory and physicochemical characteristics of EVOO extracted from olives of the Picual variety harvested in Uruguay.

### 2 MATERIALS AND METHODS

#### 2.1 Samples

The olives were harvested in five different olive plantations located in the southeastern and southwestern regions of the country during the 2021 and 2022 harvests. A sample protocol was established according to which the olives had to be collected from the four sides of the tree at different heights, starting from the external part and ending in the internal part of the tree. For each sample, the trees were divided into two blocks of six trees each. First, a representative sample of 10 kg was handpicked from both blocks. After the olives were harvested, they were immediately transferred into plastic baskets to the Abencor System pilot plant, located in the School of Chemistry (UdelaR), to avoid alterations in the fruits.

The RI of olives was determined according to a classification based on the fruit color of both skin and pulp as described in the "Método de Índice de Madurez" (Uceda & Frías, 1975). The olives were classified as green (G) when their RI values were lower than 2.0, medium (M) when the values were between 2.0 and 3.0, and ripened (R) when the values were higher than 3.0, as described by Franco et al. (2015) for olive classification.

The moisture content of olives was determined by gravimetric analysis. An amount of 20 g of the homogeneous paste of olive, milled by an Abencor System hammer mill, was weighed, placed in glass Petri dishes, and dried in a conventional oven at 105 °C for 12 h. The decrease in weight was expressed as a percentage of moisture according to Reboredo-Rodríguez et al. (2015).

The lipid content of olives was determined on the same paste, milled by the Abencor System hammer mill. The paste was dried in a conventional oven at 105 °C for 12 h, and the oil was extracted in a Soxhlet apparatus (Quimis aparelhos Cientificos Ltda. Q.308.26) using petroleum ether (62–68 °C boiling point) as a solvent. The extraction was performed for 8 h. Afterward, the solvent was removed with a rotary evaporator until a constant weight was obtained (AOAC Method 945.16).

## **2.2** Extraction through the Abencor System of the Laboratory Plant

Virgin olive oil extraction of collected olives was conducted in the laboratory plant of the School of Chemistry within 24 h of harvesting as suggested by Reboredo-Rodríguez et al. (2015). The extraction conditions were as follows: the sieve size of the hammer mill was 5 mm and the olives were crushed at 3,000 rpm. Previous studies (Ellis, 2016) showed that Uruguayan olive pastes of the Picual variety have a moisture level higher than 50%, , designating them as "difficult pastes" (Cruz et al., 2007). To facilitate the separation between the oil and other compounds of the olive paste and thus increase the extraction yield, a 2% micronized natural talcum powder was added to the olive paste during the kneading stage (Aguilera et al., 2010). The paste with the talcum powder was kneaded for 50 min at 30 °C, and during the process, 200 mL of water was added. After the kneading stage, the content of each of the eight recipients of the thermoblender was centrifuged in an Abencor vertical centrifuge at 3,500 rpm for 1 min. The yield obtained from the extraction process in the Abencor plant was calculated according to Criado et al. (2007). The extracted oil was stored and protected from light in amber glass bottles at 4 °C until analysis.

#### 2.3 Chemical characterization of the oils

#### 2.3.1 Free acidity

Free acidity was determined according to the International Olive Council (IOC) guideline, IOC/T.20/Doc. No 34/Rev.1 (IOC, 2017), and was expressed as the percentage of oleic acid.

#### 2.3.2 Determination of the total content of phenolic compounds

The total phenolic content was determined according to the IOC/T 20/Doc. No 29/Rev.2 (IOC, 2022b) method. The extraction was performed using 2.0 g of EVOO and methanol/water 80:20 V/V solution, together with the addition of a syringic acid solution. For the determination, an HPLC Shimadzu 20 A was used together with a diode array detector (SPD M20A model) and a Phenomenex C18 column (4.6 mm diameter, 25 cm length, and 5  $\mu$ m particle size). The phenolic compounds were quantified at a 280-nm wavelength using water with 0.2% of H<sub>3</sub>PO<sub>4</sub>/methanol/acetonitrile 96:2:2 V/V/V as a mobile phase, according to the COI 2022 recommendation. The external calibration standards were tyrosol and syringic acid.

#### 2.3.3 Determination of the content of total tocopherols

The total tocopherols were quantified by HPLC using acetonitrile, methanol, isopropyl, and water as the mobile phase under conditions described in the study of Andrikopoulos et al. (1991). The oil extracts dissolved in isopropyl were quantified in a chromatograph (Shimadzu 20 A HPLC) coupled with a fluorescence detector (Shimadzu RF 20 A XS) with the excitation and emission wavelengths at 290 and 330 nm, respectively. A Macherey-Nagel C18 ( $250 \times 4.6$  mm,  $100 \mu$ m) column packed at 40 °C was used.

#### 2.3.4 Fatty acid composition

The fatty acid profile of oil extracts was analyzed by gas chromatography (GC) on a Shimadzu GC-14B (Kyoto, Japan), equipped with a FID and a capillary column SP 2330 (30 m ' 0.25 mm ' 0.2 um). The temperature program was as follows: initial temperature 160°C, increased at a heating rate of 4°C/min to 230°C, and held at this temperature for 10 min. Fatty acid methyl esters were prepared according to the International Union of Pure and Applied Chemistry (1987) and analyzed by GC (according to the AOCS Ce 1c-89 and AOCS, 1998). Peak identification was accomplished by the analysis of authentic standards.

All chemical analyses were done in triplicate.

#### 2.4 Sensory characterization of oils

Commercial quality and descriptive profile were obtained through descriptive analysis with a sensory panel of 10 judges. The judges were recruited among teachers and staff of the School of Chemistry (Universidad de la República), chosen according to the IOC (IOC, 2021) guidelines, and received more than 200 h of training in the descriptive analysis of olive oil. The panel has been recognized by the IOC since 2013.

The samples were evaluated in duplicate in two sessions. The samples were presented one by one in a sequential monadic manner, in an aleatory order, and encoded with three-digit numbers. The tasters evaluated 14-16 mL of each oil in blue glass cups (IOC, 2020) at  $28 \pm 2$ °C. The samples were measured with unstructured numerical intensity scales of 10 cm with the ends "none-too much," negative attributes or defects (fusty/muddy, musty/humid/earthy, winey/vinegary/acid/sour, frostbitten olives (wet wood), rancid, and others), and positive attributes (fruity, bitter, and pungent) (IOC, 2018). Moreover, the following positive attributes were added: green leaf/herb/grass, fig, tomato, apple, banana, almonds/dry fruit, sweet, and astringent (Bongartz & Oberg, 2011; Jiménez et al., 2013).

The samples were evaluated under the conditions described in the IOC (2018) guidelines, in a standardized taste room according to the IOC/T.20/Doc. No 6/Rev. 1 (IOC, 2007), provided with five individual cabins, with a controlled temperature (between 22 and 24°C) and air circulation.

#### 2.5 Statistical analysis

Physicochemical and sensory data were analyzed by the analysis of variance (ANOVA) using RI, crop year, and the interaction crop year × RI as fixed sources of variation, followed by the Tukey test to compare the means that showed significant variation (p < 0.05). Then, principal component analysis (PCA) was carried out with the variables measured. Statistical analyses were performed using the XL Stat 2021.7 software (Addinsoft, NY, USA).

#### **3 RESULTS**

#### 3.1 Chemical characteristics of olives and extraction yield.

A total of 19 samples of olive oils were analyzed. They were obtained from olives of two different harvests (2021 and 2022): four samples of green olives (G) with RI between 1.5 and 1.8 (two of 2021 and two of 2022 harvests), six samples of medium olives (M) with RI between 2.2 and 3.0 (two of 2021 and four of 2022 harvests), and nine samples of ripened olives (R) with RI between 3.1 and 4.1 (eight of 2021 and one of 2022 harvests).

The moisture content of the olives was not significantly affected by the harvest year (p = 0.1992), being at values between 55.9 and 66.9%, which confirms what Ellis (2016) reported that Uruguayan olive pastes of the Picual variety are considered "difficult pastes." Aguilera et al. (2010) characterized Spanish Picual olive pastes as "very difficult pastes" with a moisture value of 56.3%.

The RI significantly affected (p = 0.0063) the moisture content of olives. The ripened olives (R) had a higher moisture content (64.6%) than green olives (G) and medium olives (M) (60.1 and 61.7%, respectively).

The high moisture content of olives was reflected in Abencor's yield of extraction, between 9.6 and 12.9%. The harvest year did not significantly influence Abencor's yield (p = 0.0944). Neither the RI (p = 0.1569) did. Although Franco et al. (2015) suggested that there is a higher yield in olives with a higher RI due to the water loss in the olives, while the lipid biosynthesis slowly continues during the ripening process, the highest moisture content of the ripened olives analyzed did not allow an increase in the extraction yield.

The highest moisture content of the analyzed ripened olives was reflected in the content of lipids based on wet weight, being significantly lower (p = 0.0222) for ripened olives (13.7%) than for green and medium olives (14.9 and 16.0%, respectively). But the content of lipids based on dry weight was not influenced by the RI, being in the same range from 37.7 to 42.1%. The results are shown in Table 1. The values reported are the mean values of the triplicates made.

Ripening index (RI)	Year	Moisture (%)	Abencor System Yield (%)	Oil yield (% wet weight)	Oil yield (% dry weight)
V	2021	59.9 a	12.9 a	16.2 b	40.8 a
V	2022	60.3 a,b	11.1 a	13.7 a,b	34.6 a
М	2021	60.7 a,b	12.4 a	16.6 b	42.6 a
М	2022	62.7 a,b	12.0 a	15.6 a,b	41.6 a
R	2021	63.7 a,b	11.3 a	15.2 a,b	42.1 a
R	2022	65.4 b	9.6 a	12.1 a	35.1 a
p-value		0.0203	0.3598	0.0272	0.0537

 Table 1. Results of the olive properties.

Means with a common letter in the same column are not significantly different (P > 0.05) according to the Tukey test.

#### 3.2 Chemical characterization of oils

#### 3.2.1 Free acidity

The percentage of free acidity is a direct measure of the quality of olive oil and represents the extension of the hydrolytic activities that reflect the care taken from the blossoming and fruit set to the probable sale and consumption of the oil (Reboredo-Rodríguez et al., 2016). The year of harvest and the RI did not significantly influence the free acidity (p > 0.05), being all the values below 0.2%. Other authors have reported that in other olive varieties, the values of free acidity increased as the RI increased due to its higher enzymatic activity, particularly lipolytic enzymes, higher sensitivity to mechanic damage, and infections due to pathogens (Franco et al., 2015); however, this was not confirmed in our study.

All oil samples that were analyzed showed free acidity values lower than 0.8%, which is the upper limit established by the IOC (2022a) for an oil to be categorized as EVOO (Table 2). This shows that the olives used in the present study were in good condition at the moment of oil extraction, and the extraction itself and subsequent storage were effectively performed (Rodrigues et al., 2018).

# 3.2.2 Determination of the total content of phenolic compounds

According to Katsoyannos et al. (2015), as the RI increases, the total content of polyphenols decreases. As shown in Table 2, this was confirmed in the present study, presenting the oils extracted from ripened olives having a significantly lower content of polyphenols (p = 0.0005) than green and medium olives. In our study, the year of harvest did not significantly influence the content of polyphenols in the oils (p = 0.0612).

#### 3.2.3 Determination of the content of total tocopherols

According to Reboredo-Rodríguez et al. (2016), tocopherols are the main lipid-soluble antioxidants present in the EVOO. Four isomers can be found in the EVOO ( $\alpha$ ,  $\beta$ ,  $\gamma$ , and  $\delta$ ), with  $\alpha$ -tocopherol (vitamin E) as the most abundant. Table 2 lists the values obtained for tocopherols in this study. There were no significant differences in the total content of tocopherol for different RI values (p = 0.0622) and for different years of harvest (p = 0.7221).

Ripening index (RI)	Year	Free acidity (%)	Polyphenols (ppm)	Tocopherols (ppm)	
V	2021	0.15 a	367.0 b	162.0 a	
V	2022	0.15 a	309.5 a,b	155.0 a	
М	2021	0.14 a	337.5 b	182.0 a	
М	2022	0.17 a	232.8 a,b	158.8 a	
R	2021	0.17 a	188.3 a	166.3 a	
R	2022	0.18 a	191.0 a	186.0 a	
p-value		0.7443	< 0.0001	0.0796	

Means with a common letter in the same column are not significantly different (P > 0.05) according to the Tukey test.

#### 3.2.4 Fatty acid composition

As shown in Table 3, the year of harvest significantly affected the contents of some fatty acids (p < 0.001), even though slightly, having the olives of 2021 harvest lower content of 16:0, 16:1, and 18:2 and higher content of 18:1 than the olives of 2022.

The oleic/linoleic ratio was significantly affected by the year of harvest (p = 0.0003), as well as for the RI (p < 0.0001). The oils of 2021 had a higher oleic/linoleic ratio than the ones of 2022 (26.0 vs 22.6). The oleic/linoleic ratio decreased with olive ripening; thus, the oils obtained from the green olives, the medium olives, and the ripened olives had values of 29.0, 24.9, and 18.9, respectively.

The harvest year influenced the unsaturated/saturated ratio (p = 0.0149). The oils extracted in 2021 had a higher ratio than those extracted in 2022 (5.8 vs 5.4).

According to Montaño et al. (2016), the composition of fatty acids depends on olive varieties. This is a decisive factor for the composition of the different fatty acids. The main fatty acid in all cases was the oleic acid (between 77.1 and 79.9%), followed by the palmitic, linoleic, and stearic acids. The Picual variety is characterized by a high content of oleic acid and a low content of linoleic acid.

With the data analyzed and taking into account the ripening stage of olives, a clear variability is present in the different fatty acids of the oils from green olives versus ripened olives. There is a decrease in the levels of oleic acid and also slight increases in linoleic and palmitic acids.

In addition, as the RI of olives increases, the ratio of the percentages of oleic/linoleic acids in the oil decreases.

#### 3.3 Sensory characterization of oils

In the 19 samples of VOO evaluated, the panel of sensory judges did not perceive defects and perceived a positive sensory attribute such as fruity; thus, according to the IOC regulations, all olive oils were classified as extra virgin.

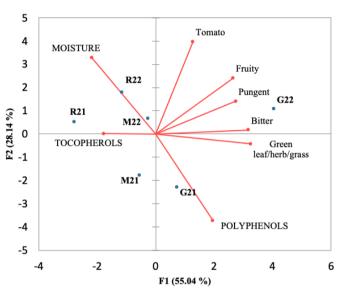
As shown Table 4, in the evaluated samples, the sensory panel perceived the attributes such as fruity, bitter, pungent, green, banana, dry fruit, and astringent. According to the results of the analysis of variance, the interaction year × RI was not significant in any of the sensory attributes (p > 0.05), which allows us to study the effect of each factor independently.

All the evaluated oils presented similar aromatic notes to banana (between 1.2 and 2.0) and dry fruit (almond and nut) (between 1.7 and 1.9) and astringent flavor (between 0.8 and 1.3). The year of harvest significantly affected the fruity (p < 0.0001), bitter (p = 0.0273), pungent (p = 0.0008), green (p < 0.0010), and tomato intensity (p = 0.0267). The oils of 2021 had lower intensities of all these attributes than the oils of 2022. The RI significantly affected the fruity (p = 0.0289), bitter (p = 0.0001), pungent (p = 0.0097), and green intensities (p = 0.0356). As shown in Table 4, the green oils presented higher intensities of all these attributes than the oils obtained from the medium and ripened olives.

A principal component analysis (PCA) with the main physicochemical and sensory data was performed. The first two main components represented 83.2% of the variance. As shown in Figure 1, the first main component (F1) was positively related to the fruity, bitter, pungent, and green sensory attributes and the content of phenols, but negatively related to the content of tocopherols and moisture. The second main component (F2) was positively related to moisture and tomato sensory attribute,

Table 3. Results of the composition of fatty acids from the extracted oils.

amples, the sensory<br/>ity, bitter, pungent,<br/>according to the re-<br/>ction year  $\times$  RI was<br/>tes (p > 0.05), which**4 DISCUSSION4 DISCUSSION**<br/>The Abencor yield of oils extracted from Uruguayan Picual<br/>olives was lower (between 9.6 and 12.9%) than that reported<br/>from Spanish Picual, due to the high moisture of the olives (55%)



but negatively related to the content of total phenols. A strong correlation is observed among fruity, pungent, bitter, and green

sensory attributes which decrease as samples increase ripening.

On the contrary, an inverse correlation is observed between the

moisture and the content of phenols in the samples.

Figure 1. Principal component analysis carried out on sensory and chemical data results.

Ripening Index (RI)	Year	16:0	16:1	18:0	18:1	18:2	18:3	20:0	20:1	24:0	Oleic/linoleic acids	Unsaturated/ saturated ratio
V	2021	11.65 a	0.90 a	2.20 a	79.90 c	2.65 a	0.65 a	0.30 a	0.25 a	0.10 a	30.46 d	5.93 a
V	2022	12.95 b	1.25 b	2.10 a	78.25a,b,c	2.85 a,b	0.55 a	0.30 a	0.20 a	0.10 a	27.58 d	5.41 a
М	2021	11.90a,b	1.00 a,b	2.20 a	79.55b,c	2.95 a,b	0.60 a	0.30 a	0.20 a	0.10 a	27.19 c,d	5.83 a
М	2022	12.58a,b	1.30 b	2.60 a	78.10a,b	3.48 b,c	0.58 a	0.30 a	0.20 a	0.10 a	22.69 b,c	5.26 a
R	2021	12.41a,b	1.26 b	2.39 a	77.80 a	3.90 c,d	0.63 a	0.30 a	0.20 a	0.10 a	20.28 a,b	5.56 a
R	2022	12.50a,b	1.30 b	2.30 a	77.10 a	4.40 d	0.60 a	0.30 a	0.20 a	0.10 a	17.52 a	5.50 a
p-value		0.0091	0.0001	0.6288	0.0001	< 0.0001	0.1944	0.9999	0.1245	0.9999	< 0.0001	0.0609

Means with a common letter in the same column are not significantly different (P > 0.05) according to the Tukey test.

Table 4. Results of the sensory	analysis of the EVOOs.
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Ripening index (RI)	Year	Fruity	Bitter	Pungent	Green	Tomato	Banana	Dry fruit	Astringent
V	2021	4.0 a	3.2 a,b	3.2 a,b,c	2.7 a,b	1.3 a	1.8 a	1.9 a	1.3 a
V	2022	4.8 b	4.0 b	4.1 c	3.1 b	2.4 b	2.0 a	1.7 a	1.0 a
М	2021	3.7 a	2.9 a	2.9 a,b	2.7 a,b	1.6 a,b	1.7 a	1.8 a	1.1 a
М	2022	4.4 a,b	2.7 a	2.9 a,b	2.6 a,b	1.9 a,b	1.7 a	1.9 a	1.0 a
R	2021	3.8 a	2.2 a	2.3 a	2.3 a	1.9 a,b	1.6 a	1.8 a	0.8 a
R	2022	4.1 a	3.0 a	3.6 b,c	2.5 a,b	2.0 a,b	1.2 a	1.9 a	1.2 a
p-value		< 0.0001	< 0.0001	< 0.0001	0.0013	0.0400	0.5846	0.9730	0.3648

Means with a common letter in the same column are not significantly different (P > 0.05) according to the Tukey test.

higher). For example, Gallardo González (2015) reported that the Abencor yield obtained from Spanish olives with a very low RI (0.21) was 10.7%, whereas the yield reached 20.6% in olives with an RI higher than 4.

The appearance of difficult pastes depends on both the moisture content and the quantity of interference compounds (colloids and surfactants) that are related to the degree of fruit ripening, crop conditions, and the variety of olives (Cruz et al., 2007). These pastes are characterized by a low yield of extraction and a great content of remnant oil in the pulp after centrifugation, causing losses to the producer because, during the milling phase, stronger emulsions are formed, which are impossible to break under kneading conditions.

The phenolic compounds are considered natural antioxidants responsible for the oil stability through oxidation and contribute to sensory characteristics such as bitter and pungent flavors (Reboredo-Rodríguez et al., 2016). The content of phenols of the studied oils (between 188.3 and 367.0 ppm) was similar to that reported by other authors for the Spanish Picual variety. For example, Montaño et al. (2016) reported a content of phenols of 380.7 ppm in oils of the Extremadura region. Gallardo González (2015) reported a content of phenols of 286.1 ppm in oils from the same region. According to Tous et al. (1997), the Picual variety can be classified as "High content of total phenols."

According to Jiménez et al. (2013), during the fruit ripening process, a series of metabolic processes (chemical and enzymatic reactions) occur, resulting in the production of free phenols and the induction of variations in the phenolic profile of several compounds. These changes affect the quality, sensory properties, oxidative stability, and/or nutritional value of the oil obtained. The decrease in phenol content during ripening that was found in this study was also reported by other authors (Gallardo González, 2015).

The tocopherols have the activity of elimination of free radicals both *in vivo* (vitamin E) and *in vitro*. Therefore, they contribute to giving oxidative stability to the oil and play a beneficial role in health due to their antioxidant activity. The major tocopherol is  $\alpha$ -tocopherol, representing 95% of the total of tocopherols in VOO, and considered to be the most biologically active as vitamin E.  $\beta$  and  $\gamma$  forms are below 10%, while  $\delta$  form is at very low proportions.

The tocopherol content found was between 155 and 186 mg/kg, which is in agreement with López-Cortés et al. (2013) who reported the  $\alpha$ -tocopherol content between 131.5 and 269.9 mg/kg in oils obtained from Alfafara, Farga, Morruda, and Picual varieties of olives cultivated in the east of Spain. It should be considered that hydrophilic antioxidants and polar phenols in an oil matrix remain oriented to the interphase air–oil, turning to be more protective against oxidation than the lipophilic antioxidants such as the tocopherols which remain within the matrix (Frankel, 1996). Therefore, the polyphenols will give the greatest protection against the oxidation processes.

The tocopherols are also important for their nutritional value. According to Rodrigues et al. (2021), Arbosana, Picual, and Cornicabra varieties of olive oils have a high number of tocopherols, which gives them a high nutritional value, and their consumption would be beneficial for health.

The fatty acid composition of olive oils is composed of monounsaturated fatty acids, specifically oleic acid (55–83%), saturated, mainly palmitic (7.5–20%) and stearic acids (0.5–5.0%), and polyunsaturated such as linoleic (3.5–21%) and linolenic acids (0–1.5%). These values vary among other factors according to the olive variety, climate conditions, extraction system, and ripening stage of the olive. Ranks are established by the IOC.

The oleic acid is found in high proportion in the analyzed oils with values above 77%, which is a characteristic of the Picual variety and matches with what Montaño et al. (2016) reported. Rondanini et al. (2011) established a classification of varieties based on their content of oleic fatty acid. Varieties with below 55% were classified as "low oleic variety", an intermediate situation between 55 and 65% "mid-oleic variety", and lastly, varieties above 65% "high oleic variety". In our study, the Uruguayan Picual variety is a "high oleic variety."

The content of high oleic acid, which is responsible for providing high oxidative stability to the oil, contributes to a lower incidence of chronic diseases for the population. This is because oleic acid reduces the content of low-density lipoprotein, increases the content of high-density lipoprotein, and reduces the content of triglycerides in the blood, in addition to having a hypotensive effect (Guo et al., 2018).

The linoleic acid (between 2.6 and 4.4%) is the higher proportion of polyunsaturated fatty acid, followed by the linolenic acid with contents far below the linoleic acid, below 1%. Regarding the implication of linoleic and linolenic fatty acids, their presence is essential not only from the nutritional point of view (essential for the human diet) but also for their contribution to the "green fruity" and "bitter" attributes of the oils. Moreover, the compounds responsible for these attributes are produced from these fatty acids through the lipoxygenase route (Diraman et al., 2011). The main saturated fatty acid is the palmitic one with higher values of 11.6% in all samples. This is in concordance with what Gallardo González (2015) reported on the oils obtained from olives from Extremadura (Spain).

The oxidative stability of olive oils is very influenced by their natural antioxidant content (polyphenols and tocopherols) and their percentage of monounsaturated and polyunsaturated fatty acids. The higher the oleic/linoleic ratio or the lower the unsaturated/saturated ratio, the more stable will be the oil during the time (longer shelf time). Thus, in the studied oils, because of their high percentage of oleic and low percentage of linoleic, they have a high oleic/linoleic ratio, which, however, significantly decreases when the RI increases.

The sensory profile of Uruguayan olive oils of the Picual variety did not wholly match with what other authors reported for the same variety of olives. There are few reports on the sensory profile of oils extracted from Picual olives, and the results varied according to the crop region and the year of harvest. For example, Diarte et al. (2021) studied oils extracted from Picual olives cultivated in Cataluña Province (Spain), from the area Protected Designation of Origin "Siurana" with 2.3 RI, reporting fruity, bitter, and pungent values which are higher than those of Uruguayan oils (6.2, 5.2, and 5.2, respectively). These authors also report that the oxidative stability of Picual oils assessed through the Rancimat method was the highest of all the studied varieties. These data might be related to the content of phenolic compounds which improve the oxidative stability (Vázquez Roncero et al., 1975); this would, therefore, explain the highest values of bitter and pungent perceived in these oils.

However, Jiménez Herrera et al. (2012) reported lower values of bitter (2.4–4.5) and pungent (2.2–3.1) in oils extracted from olives of the Picual variety from Córdoba Province (Spain), similar values that were found in Uruguayan oils. The intensity of the fruity attribute in those oils was higher (5.7–6.2) than that detected in our study. In those oils, aromatic notes of green leaf, green herb, almond, apple, and fig (the last one is very typical from the Spanish Picual olive oils) were also perceived. It is to mention that in any of the studied Uruguayan oils notes of apple and fig were perceived, but, notes of green, dry fruit, banana, and tomato were not.

Baena González (2018) sensorily analyzed two samples of oil extracted from Picual olives of different harvests from Granada Province (Spain), reporting fruity of 3.5 in green and ripened olives, bitter of 2.5 and pungent of 3.0 in green olives, bitter of 1.0, and pungent of 0.5 in ripened olives, the values of which were lower than those found in our study.

Other authors such as Sánchez Casas et al. (2006) sensorily characterized 17 samples of oils extracted from olives of Picual varieties cultivated in the Province of Extremadura (Spain) in three ripening stages: green, medium, and ripened. They reported that the Picual variety presents an "intense fruity with a bitter and pungent flavor," but having used a scale with a five-point amplitude, the results are not comparable with ours.

The sensory qualities of VOO are affected by the presence of specific lower compounds, among which the phenolic compounds are the most important. The phenolic compounds are responsible for the flavor characteristics such as bitter, pungent, and astringent. They exhibit an antioxidant activity that is widely responsible for the oxidative stability, shelf life, and nutritional value of VOO (Lukić et al., 2017; Xiang et al., 2017). In the present study, the relationship between the sensory characteristics and the polyphenol content was confirmed in the studied samples because the presence of phenolic compounds stimulates the taste receptors and the trigeminal nerve, perceiving the sensations of bitter in the first case and the pungent and astringent in the second case.

An increase in RI of the olives resulted in a decrease in bitter, pungent, and green sensory attributes in oils, which matches with the information reported by Jiménez Herrera et al. (2012). These authors sensorily characterized the oils extracted from olives of the Picual variety from Córdoba Province (Spain), with RI between 0.4 and 4.7. They detected that the oils obtained from collected olives in the lowest ripening stage has higher and statistically significant values in the bitter and pungent attributes, for the ones obtained from the oils with higher ripening stages. Regarding the "fruity" attribute, they highlight that this attribute reached the highest values of intensity in the samples with RI between 1.2 and 2.2, with a maximum score of 7.2. These results agree with those of Rotondi et al.'s (2004) study, regarding the varieties of Italian olives that show how the "fruity" attribute reaches the maximum values in lower levels of ripening.

These sensory characteristics of the Uruguayan Picual oils make this variety especially suitable for consumers not accustomed to the flavor and aroma of intense virgin olive oils like Coratina variety.

## **5 CONCLUSIONS**

All the studied pastes were considered "difficult pastes" due to the high moisture content in Uruguayan olives, Picual variety. This was reflected in low extraction yields (despite the addition of 2% micronized talc powder during the kneading stage). This implies a great content of remnant oil in the olive pomace, causing high losses to the producer. As future trends, the application of other technological coadjuvants (calcium carbonate, enzymes) and/or the use of new technologies (ultrasound, megasound, microwaves, etc.) might be studied to increase the extraction yield.

The content of total phenols and oleic acid allows defining the Uruguayan Picual oils as "high in total phenols" and "high in oleic acid." They were sensory characterized as oils with medium fruity, medium bitter, and medium pungent and with green, tomato, banana, and dry fruit aromatic notes. The fruity, bitter, and pungent, polyphenol content, the oleic content, and the oleic/linoleic ratio decreased in the oils obtained from ripened olives, without affecting the yield. This is what makes the Uruguayan olive oil of the Picual variety ideal to be harvested with low RIs.

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