

Factors Affecting the “Spanish Green Olive” Process: Their Influence on Final Texture and Industrial Losses

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Several factors affecting “Spanish green olive” processing (fruit ripeness, fruit size, lye concentration, and its penetration through the flesh) have been studied at industrial and/or pilot plant scales, the final texture being related to pitting losses and cell wall composition. Olives at different stages of ripeness suffered major losses during the pitting process: green unripe olives were excessively firm and the cell wall of changing-color olives was too disorganized to give good industrial results. When the other factors were studied, a very clear result was obtained: only in very strong treatments (high concentration of lye and 100% penetration into the flesh), whatever the fruit size, were major losses quantified. Furthermore, these losses were related to significant decreases in texture and to changes in the composition of polysaccharides, which are very important in the cell wall structure: carbonate-soluble pectins, 24% KOH-soluble hemicelluloses, and α -cellulose.

Keywords: Olive; ripening; processing; texture; pitting losses

INTRODUCTION

The “Spanish green olive” style is one of the most esteemed among all table olive styles for the organoleptic characteristics of the final product. This processing consists of an initial treatment with diluted sodium hydroxide (lye), followed by a washing step to eliminate most of the sodium hydroxide from the flesh. Subsequently, the fruits are placed into a solution of sodium chloride (brine), where a lactic fermentation takes place. A process so complicated has many factors that could influence the final quality of the table olives. Several of these factors have been studied for several years, i.e. lye concentration and temperature of the treatment (Rodríguez de la Borbolla, 1981; Sánchez et al., 1990) or different washing processes (Rodríguez de la Borbolla and Rejano, 1978; Sánchez et al., 1995). Even a rapid process with lye treatment but without fermentation has been studied to allow the industry to pack the olives in a very short period of time, the olives having a taste very similar to those from the traditional process (Montaño et al., 1986, 1988). Some of these new processes have been tested in several industries, having a variable applicability. However, very little work has been done on the influence of the harvesting period or the effect of variations from the traditional process on the economic losses of the industrial producers (Montero-Marín and Fernández-Díez, 1976). In addition, it is important to stress that nowadays producers are very interested in the quality of their table olives owing to the increasing competitiveness, better olives leading to higher consumer acceptability.

Texture is the organoleptic characteristic that is most directly related to product losses in the industry and one of the main reasons for consumer rejection. As has been pointed out in previous studies (Jiménez et al., 1995, 1996), there are very clear relations between processing, firmness changes, and cell wall structure. For this reason, the aim of this work is to determine the implications of several processing factors (fruit

ripeness, fruit size, lye concentration, and its penetration through the flesh) on olive firmness and on industrial losses, trying to relate these results to changes in the composition of cell wall polysaccharides. Determining the fundamental reason for the textural changes could help in the improvement of olive table quality and industrial profits.

MATERIALS AND METHODS

Fresh Material. Olive fruits, cv. Manzanilla (*Olea europaea pomiformis*), were harvested in different areas in the province of Seville and at different dates (from the beginning of September to the end of October) to obtain the three stages of ripeness usually utilized in the Spanish green olive processing: green unripe (GU), green ripe (GR), and changing color (CC). Fresh olives were size-sorted; only sizes 240–280 [between 240 and 280 fruits/kg (S240)], and 340–380 [between 340 and 380 fruits/kg (S340)] were chosen to study the influence of size.

Olive Processing. The study of the influence of ripeness was performed on the three stages at industrial level in 15 000 L containers. To avoid the differences between olives located at different depths in the container, samples during processing were taken from three levels: the bottom, the middle, and the top of the container. Each sample was size-sorted, the same sizes (S240 and S340) again being selected. The sodium hydroxide concentration of the lye varied depending on the stage of ripeness, being higher as the ripening process progressed: 2.11% for GU, 2.16% for GR, and 2.22% for CC. The lye treatment, washing, and fermentation were monitored by the industry staff.

The processing conditions were studied only with GR olives (both sizes) in a pilot plant in the Department of Food Biotechnology of the Instituto de la Grasa (Seville). Ten liter containers were used in these experiments. Conditions are summarized in Table 1. Each experiment was performed in duplicate. The degree of penetration was checked by cutting 10 olives transversely; the lye-penetrated area darkens within seconds and can be measured easily. Washing procedures were the same for industrial and pilot plant experiments: a first short wash (2 h) and a second long wash (12 h). After lye treatment and washes, most of the experiments developed a spontaneous lactic fermentation but some of them (experiments 4, 5, 7, and 10) needed a starter treatment: the containers were placed (Nov 24) in a thermostatically controlled room at 25 °C, and 30 g of glucose and 200 mL of an active inoculum (“starter”) were added. After a month (Dec

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Table 1. Different Conditions of Olive Processing in Green Ripe Stage

treatment	NaOH concn (%)	size	lye penetrn (%)
1	2.1	240–280	50
2	2.1	240–280	66.6
3	2.1	240–280	100
4	2.1	340–380	50
5	2.1	340–380	66.6
6	2.1	340–380	100
7	2.4	240–280	50
8	2.4	240–280	66.6
9	2.4	240–280	100
10	2.4	340–380	50
11	2.4	340–380	66.6
12	2.4	340–380	100

22), treatments 5 and 7 developed a normal fermentation but treatments 4 and 10 did not. Half the brine of these last containers was changed for clean brine and the same amounts of glucose and starter were added. Thereafter, the fermentation was normal and complete.

Firmness Determination. The fruit texture was measured using an Instron Model 1011 texturometer fitted with a Kramer shear-press cell. The operating speed was set at 200 mm/min, and the force scale was 0–500 N. The results given are the means of 15 replicates, 4.5–6 g each.

Determination of Fermentation Parameters. The parameters studied were pH, free acidity (the sum of different free organic acids present in the brine, expressed as grams of lactic acid per 100 mL of brine), and combined acidity or “residual lye” (sodium salts of free organic acids present in the brine, mainly acetates and lactates, expressed as equivalents of NaOH per liter of brine) (International Olive Oil Council, 1990). The determination of these three parameters was performed in a 670 Titroprocessor from Metrohm according to the method of Sánchez et al. (1995).

Pitting Experiences. One kilogram of each sample was pitted by both automatic (Sadrym, Model DU, Seville) and manual means, the pits being recovered. The broken olives were quantified (Montero-Marín and Fernández-Díez, 1976) as were the pits released with flesh adhered to them, and the results are expressed as percent of the total fruits per kilogram. Both percentages were considered as good measures for industrial losses, since broken olives are directly rejected before packing and the pits with flesh lead to a decrease in the net weight of the industrial production.

Cell Wall Isolation and Fractionation. To obtain cell wall material, olive flesh was treated with phosphate buffer, sodium laurylsulfate, phenol/acetic acid/water, and acetone. The different polysaccharides were fractionated from cell wall, with imidazole, sodium carbonate, 4% KOH, and 24% KOH (Sánchez-Romero et al., 1997). The fractions analyzed were as follows: phosphate-soluble fraction (PSF), imidazole-soluble fraction (ISF), carbonate-soluble fraction (CSF), 4% KOH-soluble fraction (K1SF), 24% KOH-soluble fraction (K2SF), and α -cellulose (CEL).

Polysaccharide Analysis. Neutral sugars and uronic acids of the different fractions were quantified by colorimetric means: neutral sugars by the anthrone method (Dische, 1962) and uronic acids by the phenylphenol method (Blumenkrantz and Asboe-Hansen, 1973). Absorbance values of samples and standard curves were measured in a Model 450 microplate reader from BioRad. The results are expressed as milligrams per 1000 fruits.

Statistical Analysis. The data were statistically studied by analysis of variance. Means were compared with Duncan's multiple range test ($P < 0.05$).

RESULTS AND DISCUSSION

This work started with the study of the effects of the ripeness stage (GU, GR, and CC) on the texture of fermented olives and on the losses of product during the

pitting process. At this first step, the industrial treatment was standard, varying the conditions only very slightly to adapt the process to the natural characteristics of the fruits (see Materials and Methods). In the second part of this work, where only GR fruit were processed, three factors affecting the treatment were studied: sodium hydroxide (lye) concentration, degree of penetration of the lye through the flesh, and the size of the fruits (this latter factor was related partially with the ripening process). The final losses during pitting were quantified, and the relationships between these and the different processing conditions and mainly to the final firmness values were analyzed.

Influence of the Stage of Ripeness. Fresh fruit at the different stages showed significant differences ($P < 0.05$) in texture between sizes and stages (Table 2). Firmness decreased as the ripening process progressed, its diminution being >30 N/g. From other studies on olive ripening (Heredia et al., 1993) it was concluded that it is in this period (between GU and CC) when the biggest changes in texture take place, results which agreed with those of other authors working on different olive cultivars (Fernández-Díez and Cerdón-Casanueva, 1966). Furthermore, smaller sized olives (S340) were always firmer than larger sized ones (S240) for the same stage of ripeness, as has been described for other cultivars (Fernández-Díez and Vidal-Singler, 1968; Márquez-González et al., 1975): usually, smaller olives are less ripe than bigger ones at every ripeness stage. The six samples analyzed, therefore, represented a sequence of the ripening process beginning with the smallest unripe olives and finishing with the largest changing color olives. It can be seen in Table 2 that the texture of fresh fruit decreased gradually between these two samples.

Lye treatment and fermentation almost annulled the statistical differences between textures, only the least ripe sample being different from the others. The total difference was much lower than those of fresh fruits (only 10 N/g between maximum and minimum textures). The decreases in texture because of processing were about 25% (values in parentheses in Table 2). Only the ripest sample had texture values similar to those before processing (102.16% relative texture), as happens in the case of the Gordal cultivar (Montero-Marín and Fernández-Díez, 1976). To explain this fact, it is important to bear in mind that after the decrease in texture caused by the lye treatment, there is an increase when the fruits are placed in brine (Jiménez et al., 1995), there being a recovery of around 70–80% of the initial firmness when working with green olives. This effect is due to the presence of cations (sodium and calcium) in the brine, which are able to stabilize the new structure of cell wall polysaccharides after lye treatment (Jiménez et al., 1997). The structure and characteristics of native cell wall polysaccharides in changing color olives are different from those of green fruit, the former being more sensitive to the ionic environment. This could favor a higher recovery of texture in the changing-color fruits than in the green ones.

The study of the pitting process showed significant differences between ripeness stages. The percentages of broken olives were not statistically different, unlike those of pits with adhered flesh. The samples in the middle of the ripening period under study gave the best results, the others having losses of 10–20%. The same results were found by Montero-Marín and Fernández-Díez (1976) working with Gordal olives.

Table 2. Firmness of Fresh Olives and Olives after Fermentation and Losses during Pitting Process in Three Different Stages of Ripeness: Green Unripe (GU), Green Ripe (GR), and Changing Color (CC)^a

	GU	GR	CC
(1) fresh fruit firmness (N/g)			
S240	71.48 a	64.85 b	48.84 c
S340	81.67 d	73.81 a	67.71 b
(2) fermented fruit firmness (N/g)			
S240	53.28 (74.50%) ab	49.35 (76.09%) b	49.90 (102.16%) b
S340	59.81 (73.23%) a	56.56 (76.63%) ab	48.27 (71.29%) b
(3) pitting losses (%)			
broken olives			
S240	3.11 A	0.12 A	0.53 A
S340	3.21 A	0 A	1.58 A
pits with flesh			
S240	10.87 ab	1.91 b	15.62 a
S340	20.40 a	1.78 b	17.71 a

^a In each block, numbers followed by a different letter indicate that there are significant differences between them ($P < 0.05$). In block 2, the numbers in parentheses correspond to the relative firmness of each sample referred to its initial value. In block 3 there are two comparison groups: broken olives and pits with flesh.

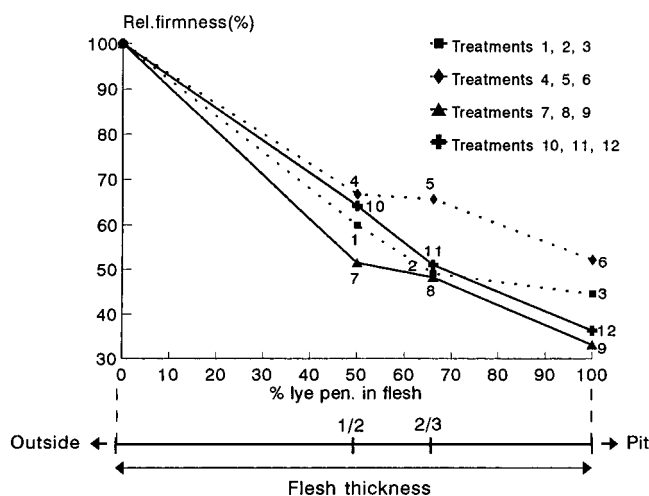


Figure 1. Firmness decreases in green ripe olives as a result of the different conditions of sodium hydroxide treatment.

Although there were no “quantitative” differences in the percentages of pits with flesh in the green unripe and changing-color olives, there were “qualitative” ones, since the ways by which the pits were released from the fruits were different. In unripe olives it seemed as if the flesh next to the pit was still “adhered” to it. The flesh in fresh fruit was excessively firm, and the lye could not produce one of its desirable effects, which is to separate the flesh from the pit (International Olive Oil Council, 1990). However, in changing-color olives the situation was completely different: the fruits simply “burst” during pitting because the flesh could not resist the punch strength. Though in this case the level of texture of fermented fruit was normal, the cell wall and middle lamella structures are disorganized by both ripening and industrial processes and, as a consequence, the pit tears away part of the flesh.

Effect of Different Lye Treatments on Texture. In Figure 1 the values of texture after lye treatment in the different conditions are presented. The following three factors affected firmness.

Size. Smaller olives were always firmer than larger ones under the same processing conditions. The differences between sizes were greater with 2.1% lye (dotted lines in Figure 1), being statistically significant in the three degrees of lye penetration. However, with 2.4% lye, they were significant only in half-penetration.

Lye Concentration. More concentrated lye produced higher losses of firmness, its effect being higher as the

lye advanced into the flesh. Lye penetrations of 2.4% and 100% (treatments 9 and 12, respectively) led to the lowest firmness values.

Lye Penetration. This factor interacted very strongly with the preceding one, mainly at the highest levels (treatments 9 and 12, as described above). The penetration of lye two-thirds of the way to the pit produced important texture losses only in the case of the 2.4% lye, the total loss of firmness being represented by an almost straight line between 0% and 100% penetration.

The only experiment that had a firmness value higher than expected was treatment 5—low lye, small size, and two-thirds penetration (Figure 1). Furthermore, in this experiment fermentation was not spontaneous, as will be described below, both events pointing to the fact that the lye probably could not totally penetrate two-thirds of the distance to the pit.

Evolution of Fermentation. At this stage of the work, the factor that had the most influence was the degree of penetration of the lye. Thus, there were differences among “underdone” (half penetration), “done” (two-thirds penetration), and “overdone” (penetration to the pit) treatments. Done and overdone olives developed a spontaneous lactic fermentation (Figure 2), there being a marked pH decrease and a marked free acidity increase a few days after the olives were placed in brine.

Only underdone experiments presented problems (treatments 4, 7, and 10) and also experiment 5, which has been described above as an irregular treatment. Experiments 5 and 7 showed slight variations from a normal fermentation and needed only one starter addition, unlike treatments 4 and 10, which needed two. The problems consisted in high pH values and low levels of free acidity 1 or 2.5 months after lye treatment, whereas the other fermentations were considered to be finished after 2.5 months (Figure 2). This delay in the fermentation was mainly due to a very low penetration of sodium hydroxide through the flesh in the case of underdone olives. Usually, an industrially correct lye treatment must penetrate to two-thirds or three-fourths of the flesh thickness, and in these cases it reached only half. Furthermore, it seemed as if smaller olives were more difficult to ferment since experiments with half-penetration and larger olives (experiments 1 and 7) needed no or only one step with starter, while the smaller ones (treatments 4 and 10) needed two. In this second treatment, the change of brine was important (see Materials and Methods) because the brine in the containers was very dark. Its content in polyphenols was high, and it is well-known that these compounds

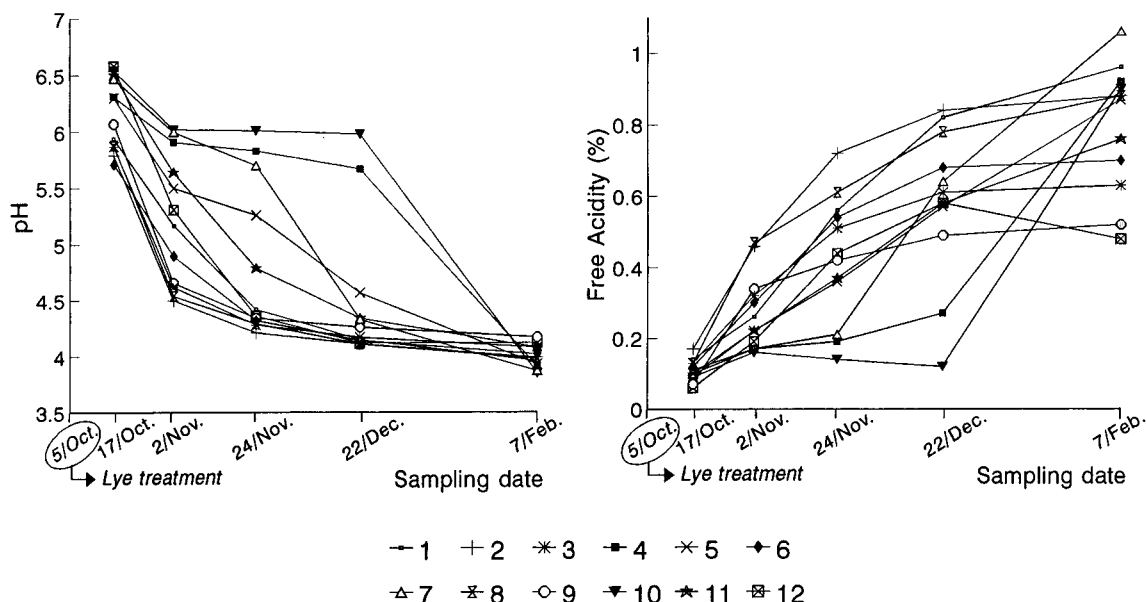


Figure 2. Evolution of two fermentation parameters (pH and free acidity) in green ripe olives under different processing conditions.

of olive pulp are very strong inhibitors of microorganism growth (Rodríguez de la Borbolla, 1981; Ruiz-Barba et al., 1990, 1991).

Experiment 5 (small size, two-thirds penetration, and low lye concentration) proved to be the exception to the above explanation. This was designed to be a normal treatment (two-thirds penetration) but it proved not to be so: delayed fermentation and firmness values higher than usual after lye treatment (as described above) were two clear consequences. Another factor that might have influenced this behavior was that the olives were small. As a less ripe fruit, the level of free sugars in the fresh flesh is lower and the cell wall is more strongly structured. Thus, treatment with low concentration does not seem to have enough strength to be considered a normal process. The low level of sugars released from the flesh (free sugars and/or those hydrolyzed and solubilized from cell wall polysaccharides) could have made fermentation difficult, although the lye had penetrated two-thirds of the way into the pulp.

Although the overdone treatments (experiments 3, 6, 9, and 12) developed a spontaneous fermentation, the final values of free acidity were lower than those of the others. A lye treatment with deeper penetration into the flesh could have led to more efficient washes and thus to a marked decrease in fermentable sugars (Fernández-Díez, 1985). Lye concentration also had some effect since stronger lye (treatments 9 and 12) led to the lowest level of free acidity among these four treatments. Another consequence of the higher efficacy of washes was a lower level of combined acidity (data not shown). Usually this parameter is around 0.10–0.14 equiv NaOH/L, but in the overdone treatments it was around 0.8 equiv NaOH/L. This same level of combined acidity is obtained when olives undergo very strong washes after a normal (two-thirds penetration) lye treatment (Rodríguez de la Borbolla and Rejano, 1978).

Texture after Fermentation and Pitting Experiments. In Table 3 the data of absolute texture (newtons per gram), relative texture (percent of the original), and percentage of pitting losses are presented. The three factors produced differences in texture and in the percentages of pits with flesh.

Between sizes the texture values are always statisti-

cally significant, although to avoid complicating the table excessively they have been omitted from Table 3. These differences in texture did not cause any change in pitting losses, so size is not an important factor in this industrial process. However, the other two factors (lye concentration and its penetration into the flesh) led to significant changes in texture, which had effects during the pitting process. When treatments with different lye penetrations are compared (lower case letters in Table 3), it can be seen that with low lye there were no changes in any of the parameters studied. Only when a higher concentration of lye reached the pit (treatments 9 and 12, Table 3) was a significant decrease of texture observed. As a consequence, a marked percentage of pits with flesh was quantified. The same conclusion could be reached after study of the effect of the lye penetration (upper case letters in Table 3), since significant differences in texture and pitting losses were found only in treatments with penetration to the pit (again, experiments 9 and 12).

Although there were several patterns of fermentation behavior because of the different lye treatments, it seems to have had no influence on the pitting process. Experiments with different fermentations (i.e. treatments 2 and 4) did not show significant differences in pitting losses, unlike samples with the same behavior during fermentation (i.e. treatments 3 and 9) which did. Thus, fermentation is not an important factor from this technological point of view, but is very decisive in determining the typical flavor of Spanish style olives. It is very clear that the lye treatment was the main factor affecting pitting process, and there were only marked losses in those experiments with a high concentration of lye which reached the pit. Furthermore, this is directly related to fruit firmness after fermentation. This stressed the important relationship between the final texture of a product in a technological process and its behavior during subsequent handling.

After the results obtained in the different experiments on ripeness and processing conditions were studied, it was clear that firmness is a very good parameter to evaluate the quality of olives in two important moments of Spanish processing: the selection of fresh fruit and handling after fermentation. As far as the first point is concerned, it is important to bear in mind that early

Table 3. Firmness after Fermentation (Absolute and Relative) and Losses during the Pitting Process in the Different Experiments on Green Ripe Olives^a

	treatment											
	1	2	3	4	5	6	7	8	9 ^b	10	11	12 ^b
firmness (N/g)	51.01 a A	51.84 a A	49.93 a A	60.20 a A	56.93 a A	55.51 a A	51.06 a A	52.26 a A	39.17 b B	58.43 a A	57.27 a A	48.69 b B
rel firmness (%)	85.09	86.40	82.86	85.93	81.26	79.24	85.11	87.06	65.29	83.39	81.74	69.50
pitting losses (%):												
broken olives	3.20 a A	4.01 a A	2.60 a A	1.46 a A	4.83 a A	1.61 a A	5.00 a A	4.49 a A	7.55 a B	3.92 a A	4.09 a A	7.81 a B
pits with flesh	10.01 a A	11.46 a A	11.68 a A	9.03 a A	11.63 a A	14.42 a A	8.87 a A	13.83 a A	20.90 b B	14.08 a A	16.04 a A	34.23 b B

^a Numbers with different **lower case letters**: there are significant differences, comparing only the percent of lye penetration (comparison groups: 1-3, 4-6, 7-9, 10-12). Numbers with different **upper case letters**: there are significant differences, comparing only different lye concentration (comparison groups: 1-7, 2-8, 3-9, 4-10, 5-11, 6-12). Although the differences between sizes are always significant, they have not been marked. ^b The only experiments that have significant differences.

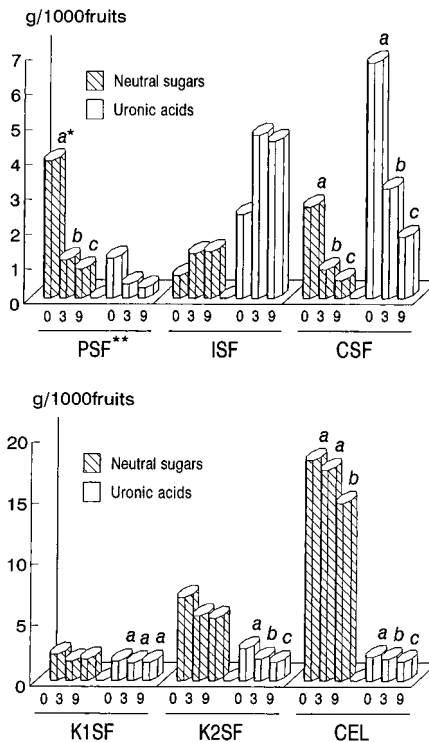


Figure 3. Composition in neutral sugars and uronic acids of cell wall fractions of three experiments (0, 3, 9). Sample 0, fresh fruit; sample 3, olives processed under treatment 3 (low losses during pitting); sample 9, olives processed under treatment 9 (high losses during pitting). *Different letters on the bars indicate significant differences. Groups of bars without letters indicate that there are significant differences only between fresh and both processed fruits.

olives suffer major losses of pulp during the pitting process owing to their high initial texture values (Montero-Marín and Fernández-Díez, 1976). As has been described above, a stronger treatment that could better separate the flesh from the pit would help to decrease these industrial losses. When changing-color olives are processed, the solution to the problem could be found in another way. These olives showed a very good response to brine sodium, their texture not decreasing after processing. Thus, the addition of other cations (mainly calcium), which have been demonstrated to have a very strong effect on increasing texture at very low concentrations (Jiménez et al., 1997), could lead to firmer olives and, therefore, to easier handling. However, olives at the appropriate stage of ripeness could also present low texture after fermentation, mainly due to very strong treatment conditions and, so, could produce losses during pitting process. This lack in firmness could be solved by the addition of cations too, as described above for changing-color olives.

Cell Wall Fractionation and Analysis. In Figure 3 the results obtained after cell wall preparation and fractionation are presented. Each fraction has been analyzed to quantify its uronic acid and neutral sugar content. As was described in previous papers (Jiménez et al., 1995, 1996), there are significant differences between fresh fruit and fermented fruit in all fractions. The aim of this step of the work was to study if there were differences between experiments that had pitting losses (treatment 9) and those that did not (treatment 3). The results, which were statistically different, mainly referred to fractions that had important implications for cell wall structure and therefore for firmness: carbonate-soluble fraction (CSF), 24% KOH-soluble

fraction (K2SF), and α -cellulose (CEL). These three fractions are the most strongly linked to the cell wall structure and are responsible for the maintenance of texture. The phosphate-soluble fraction (PSF) also showed significant differences, but it has very little relation with other cell wall polysaccharides, since it is the most soluble fraction and suffers important decreases because of processing.

It is important to point out that the general changes between fresh fruit and treatment 3 were the same as those which occurred between fresh fruit and treatment 9, the latter being more intense: a decrease in PSF, CSF, K2SF, and CEL; an increase in ISF; and almost no changes in K1SF. Only in the α -cellulose fraction was there a marked variation, since the amount of neutral sugars was not significantly different in fresh fruit and treatment 3, although it was different in treatment 9. This marked loss of cellulose could be a decisive factor in understanding the reasons for the lower firmness values and the irregular behavior during the pitting process.

In previous papers, the relationship between firmness and changes in cell wall polysaccharides has been extensively proved (Fernández-Díez, 1979; Jiménez et al., 1995, 1996). In the present study another relationship has been discussed: cell wall polysaccharides–texture–industrial behavior. The fractions that have the greatest importance for cell wall structure (pectins soluble in sodium carbonate, hemicelluloses and pectins soluble in 24% KOH, and cellulose) are those which make a difference between pitting processes with and without losses. As can be seen, the composition of the cell wall is important not only in processes so common and studied as fruit growth, ripening, and senescence but also in the efficiency of biotechnological processes. A knowledge of cell wall structure is, therefore, very important from the physiological and industrial points of view. For this reason, an increasing number of producers are interested in this subject in their search for improvement in their product quality and for the consequent higher consumer acceptability.

ABBREVIATIONS USED

GU, green unripe olive; GR, green ripe olive; CC, changing-color olive; PSF, phosphate-soluble fraction; ISF, imidazole-soluble fraction; CSF, carbonate-soluble fraction; K1SF, 4% KOH-soluble fraction; K2SF, 24% KOH-soluble fraction; CEL, α -cellulose.

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