




Effects of different comercial coatings on postharvest citrus fruit quality for export

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





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Efecto de distintos recubrimientos comerciales en la calidad poscosecha de frutos cítricos para exportación

Efeito de diferentes revestimentos comerciais na quali- dade pós-colheita de frutas cítricas para exportação

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Abstract

The use of fruit coatings aims to avoid dehydration, minimize the occurrence of spots in the peel and weight loss as well as extend the postharvest life maintaining fruit appearance. The objective of this study was to evaluate the effect of different fruit coatings in fruit quality, weight loss and acetaldehyde and ethanol production in different mandarin and orange varieties, during postharvest storage. With that aim, fruits of different varieties, with or without degreening and with postharvest fungicide treatment, were treated in experimental conditions with the commercial coatings Brillaqua UE-Mercosur and UEJ-Mercosur, Citrosol AUE and Sunseal, and Concentrol AC36, repeating the evaluation of the more promising treatments in commercial conditions together with Brillaqua UEF6-Mercosur, Citrosol AKUE y Tecnidex Teycer-GLK. In both experiments, fruit was stored for 6 weeks at 1 ± 0.5 °C plus 7 days at room temperature simulating shelf life. External and internal quality were evaluated at different moments during storage. None of the treatments affected fruit firmness, external or internal quality negatively compared with the commercial control BRIUE. The most promising treatments for their application in citrus postharvest and storage at 1 °C were Brillaqua UEF6-Mercosur, Citrosol AKUE y Tecnidex Teycer-GLK, showing an acceptable brightness level, good appearance and superior quality. Concentrol AC36 y Citrosol AUE were not discarded as alternatives since they presented acceptable brightness, appearance and acetaldehyde and ethanol levels after storage.

Keywords: storage, wax, citrus, postharvest

Resumen

El uso de recubrimientos tiene como objetivo evitar la deshidratación, minimizar la aparición de manchas en la piel y la pérdida de peso, además de prolongar la vida poscosecha y mantener la apariencia de los frutos. El objetivo del presente trabajo fue evaluar el efecto de diferentes recubrimientos en la calidad, la pérdida de peso y la producción de acetaldehído y etanol en distintas variedades de mandarina y naranja, durante el almacenamiento poscosecha. Para ello, frutos de distintas variedades, con o sin desverdizado, y con tratamiento fungicida poscosecha, fueron tratados con los recubrimientos comerciales Brillaqua UE-Mercosur y UEJ-Mercosur, Citrosol AUE y Sunseal, y Concentrol AC36, en condiciones experimentales, repitiéndose la evaluación de los más promisorios en condiciones comerciales junto con Brillaqua UEF6-Mercosur, Citrosol AKUE y Tecnidex Teycer-GLK. En ambos ensayos la fruta se almacenó durante 6 semanas a $1\pm 0,5$ °C más 7 días a temperatura ambiente, simulando la vida comercial. Se evaluó la calidad visual e interna de los frutos en diferentes momentos del almacenamiento. Ningún tratamiento afectó negativamente la firmeza, la calidad externa o interna de la fruta en comparación con el control comercial BRIUE. Los recubrimientos más promisorios para su aplicación en poscosecha de frutos cítricos y posterior almacenamiento a 1 °C fueron Brillaqua UEF6-Mercosur, Citrosol AKUE y Tecnidex Teycer-GLK, presentando un nivel de brillo, aspecto visual general y calidad superiores. No se descartan los recubrimientos Concentrol AC36 y Citrosol AUE, con aceptables niveles de brillo luego de la conservación, aspecto visual general, así como niveles de acetaldehído y etanol.

Palabras clave: almacenamiento, cera, citrus, poscosecha

Resumo

O uso de revestimentos visa evitar a desidratação, minimizar o aparecimento de manchas na pele e perda de peso, além de prolongar a vida pós-colheita, mantendo o aspecto dos frutos. O objetivo deste trabalho foi avaliar o efeito de diferentes revestimentos na qualidade, perda de peso e produção de acetaldeído e etanol, em diferentes variedades de tangerina e laranja, durante o armazenamento pós-colheita. Para isso, frutos de diferentes variedades, com ou sem esverdeamento e com tratamento fungicida pós-colheita, foram tratados com os



revestimentos comerciais Brillaqua UE-Mercosur e UEJ-Mercosur, Citrosol AUE e Sunseal e Concentrol AC36, em condições experimentais, repetindo a avaliação dos mais promissores em condições comerciais, juntamente com Brillaqua UEF6-Mercosur, Citrosol AKUE e Tecnidex Teycer-GLK. Nos dois testes, o fruto foi armazenado por 6 semanas a $1 \pm 0,5$ ° C mais 7 dias à temperatura ambiente, simulando a vida comercial. A qualidade visual e interna dos frutos foi avaliada em diferentes momentos de armazenamento. Nenhum tratamento afetou negativamente a firmeza, a qualidade externa ou interna da fruta em relação ao controle comercial BRIUE. Os revestimentos mais promissores para aplicação pós-colheita de frutas cítricas e posterior armazenamento a 1 ° C foram Brillaqua UEF6-Mercosul, Citrosol AKUE e Tecnidex Teycer-GLK, apresentando um nível de brilho, aparência visual geral e qualidade superior. Os revestimentos Concentrol AC36 e Citrosol AUE com níveis de brilho aceitáveis após a preservação, aparência visual geral, bem como os níveis de acetaldeído e etanol não são descartados.

Palavras-chave: armazenamento, cera, citros, pós-colheita

1. Introduction

Exporting fresh citrus fruit to distant markets carries the challenge of maintaining quality for extended transport periods. Fruit appearance on the market is one of the main quality factors that directly affect the price⁽¹⁾. The use of coatings, mainly waxes, with different concentration of solids aims to compensate for some of the natural waxes that are lost in the cleaning process of the fruit, avoid evaporation and transpiration loss that result in weight loss, extend fruit preservation while maintaining its firmness and freshness, and improve presentation, giving it a special shine⁽¹⁾⁽²⁾⁽³⁾⁽⁴⁾⁽⁵⁾.

The coating quality is defined by the wax content and the percentage of solids. An important factor when increasing the latter is the need for an adequate emulsion-resin ratio so as not to affect surface tension. The percentage of solids defines the coverage degree and is very important, since a poor coating (reduced level of solids) may not provide the desired final brightness and promote dehydration during storage, leading to premature aging, firmness loss and possible appearance of dehydration symptoms, spots and remarked and blackened oil glands. On the other hand, excessive coating (high content of solids) prevents gaseous exchange through the peel, reducing oxygen levels and increasing carbon dioxide levels inside, resulting in a fermentation process, product of anaerobic breathing⁽⁶⁾⁽⁷⁾⁽⁸⁾⁽⁹⁾⁽¹⁰⁾⁽¹¹⁾⁽¹²⁾. This process modifies the concentration of different volatile compounds such as ethanol and acetaldehyde, which are associated

with the appearance of unwanted flavors and odors known as off-flavors⁽²⁾⁽¹³⁾⁽¹⁴⁾⁽¹⁵⁾⁽¹⁶⁾⁽¹⁷⁾⁽¹⁸⁾.

Most citrus fruits are sensitive to low temperatures, developing different symptoms of skin damage, known as chilling injury (CI). CI symptoms do not affect the internal quality of the product but do impair its commercial quality⁽¹⁹⁾. Therefore, quarantine temperatures mandatory for export, which are close to 1°C, result in CI symptoms in sensitive varieties that reduce commercial life. The application of coatings may affect the appearance of physiological disorders. Waxing (regardless of the level of solids, 12 vs 18%) minimizes the intensity of chilling injuries, reducing their severity in different species and varieties of citrus fruits⁽²⁰⁾. In line with this, several authors determined that coatings based on shellac (E-904), carnauba (E-903), or oxidized polyethylene (E-914) prevent CI in different grapefruit varieties⁽²¹⁾⁽²²⁾. Waxing has also been found to decrease the incidence and severity of post-harvest spotting in Satsuma Okitsu mandarin⁽²³⁾.

Uruguayan citrus companies currently apply coatings based on oxidized polyethylene wax and shellac, with 18% solids, in most citrus fruits for export. However, variable environmental conditions of humidity and temperature during the packing of the fruits and the high-quality standards in the different destination markets led to the current use of coatings that do not meet companies' requirements in terms of brightness and drying efficiency.

The objective of this study was to evaluate the effect of different commercial waxes (with modifications in



their composition) on external and internal quality, weight loss, and production of acetaldehyde and ethanol in different citrus varieties: Nules Clementine, Nova, Navelina, New Hall and Salustiana, during postharvest storage. The working hypothesis focuses on the fact that changes in the composition of commercial coatings can affect the quality of citrus depending on the cultivar, and that new coatings should maintain the fruit quality and register a similar or lower weight loss than the wax currently used by Uruguayan citrus companies (BRIUE), with no negative effect on the production of compounds associated with off-flavors.

2. Material and methods

The experiment was conducted in two consecutive years in which seven different coatings were evaluated compared to the control (BRIUE). An experimental evaluation was carried out in a line for research purposes in the first year. From this first work, the most promising coatings were selected, which were evaluated again under commercial conditions (citrus packing facilities) in the following year.

2.1 Experimental trial (research line)

2.1.1 Plant material

Fruits of mandarin (*Citrus reticulata* Blanco) var. Nules Clementine, and orange [*Citrus sinensis* (L.) Osbeck] var. Navelina (both harvested on May 15) were used. They received a postharvest treatment in drencher with 1000 mg.kg⁻¹ of Pirimetanil (PIR, Fruitgard PIR-400, 400 g/l p.a. Enzur SA Uruguay), 750 mg.kg⁻¹ of propiconazole (PZ, Fruitgard PZ-100, 100 g/l p.a. Enzur SA Uruguay), 35 mg.kg⁻¹ of 2,4-D (Citrus Fix, 80 g/l p.a. Lainco SA Spain), 50 mg.kg⁻¹ of coadjuvant (Fixfilm, 20% emulsified oxidized polyethylene and 16.5% phenoxy ethoxylated alcohol, Enzur SA Uruguay) and 200 mg.kg⁻¹ of antifoam (Antifoam S7, aqueous silicone solution, Enzur SA Uruguay), and then a degreening treatment with 1 mg.kg⁻¹ of ethylene at 19°C for 72 hours and 24 hours at room temperature.

2.1.2 Application of coatings

The applications were carried out in an experimental line with manual pressure spraying

equipment with a hollow cone nozzle, collecting the fruits at the end of the drying oven (30 s at 45-55°C). The evaluated coatings were:

- BRIUE: Brillaqua UE-Mercosur [18% solids (12.20% oxidized polyethylene wax and 2.85% shellac), Enzur SA Uruguay]
- BRIUEJ: Brillaqua UEJ-Mercosur [18% solids (10% shellac), Enzur SA Uruguay]
- CITAE: Citrosol A UE [18% solids (oxidized polyethylene wax and shellac), Citrosol SA Spain]
- CITSUN: Citrosol Sunseal UE [18% solids (oxidized polyethylene wax and shellac), Citrosol SA Spain]
- AC36 (1:2 and 1:5): Frutcoat AC-36 CE dilutions 1:2 and 1:5 [36.1% solids (oxidized polyethylene wax), Productos Concentrol SA Spain]

2.1.3 Physicochemical characteristics

- Initial evaluation: At the end of the drying oven, visual observations of all the fruits of each treatment were made with emphasis on the drying of the coating and the brightness achieved, and they were weighed. Each peel color index (PCI) was evaluated, registering the values of the coordinates Hunter LAB, L, a, and b with a colorimeter (Konica Minolta CR-400, illuminant D65, Tokyo, Japan). Firmness was determined with a texture analyzer (Stable Micro Systems TA-XT plus, test probe P/75), quantifying fruit deformation (mm) when applying a force of 10 N. Fruits were processed to obtain juice using a home juicer (Skymesen, model ESB). The juice and soluble solids contents (SS, °Brix) were determined at 20°C, using a digital refractometer (Atago DBX-55, Atago Co. Ltd, Tokyo, Japan). Acidity (% citric acid) was estimated by titrating a 10 mL juice sample with 0.1 N NaOH and phenolphthalein as an indicator reagent of the change in pH, establishing the titration volume with the observation of color change at a pH of 8.1. From the relationship between SS and acidity, the maturity index was determined (MI).
- Evaluation after 3 and 6 weeks of storage, and 6 weeks + 7 days of shelf life: Fruits of each treatment were stored for 6 weeks in cold chamber at 1±0.5°C, remaining 7 more days at room



temperature between 12 and 20°C (shelf life). Net weight loss was determined each time and the incidence (number of fruits/total number per repetition*100) of rotten fruits, CI (taking into account mild, moderate, and severe damage) and symptoms of dehydration were registered. The general condition of the calyx was categorized as green, senescent, black, or absent. In addition, color, firmness, SS and acidity were evaluated.

Three juice samples of 5 mL per treatment were conserved at each evaluation moment, to determine acetaldehyde and ethanol concentration. Samples were frozen until determination moment, which was carried out by gas chromatography⁽²⁴⁾.

2.1.4 Experimental design and statistical analysis

A completely randomized design was used with five replicates per treatment. The experimental unit was 15 fruits for Nules Clementine and 12 fruits for Navelina. A random sample of 10 fruits per evaluation date was taken to evaluate PCI, firmness, juice percentage, SS and acidity. Data were transformed if necessary (square root) for variance analysis. Mean comparison was performed using the DGC test ($p \leq 0.05$)⁽²⁵⁾.

2.2 Commercial trials (citrus packing facilities)

2.2.1 Plant material

The first experiment was conducted with mandarin fruits var. Nules Clementine and orange var. New Hall (harvest April 25), previously treated in drencher with PIR (1000 mg.kg⁻¹), PZ (1500 mg.kg⁻¹) and 2,4-D (25 mg.kg⁻¹), and degreened under the same conditions described above. Subsequently, they received treatments on the packing line: bath with water and NaCl₂ (200 mg.kg⁻¹), curtain of neutral soap with 10% sodium orthophenylphenate (SOPP, Espumer-O Mercosur, 143 gL⁻¹ p.a. equivalent in orthophenylphenol), spraying with IMZ (1500 mg.kg⁻¹) and finally, waxing, where the different treatments were conducted.

In the second experiment, mandarin fruits var. Nova (*Citrus Clementine* Hort x (*Citrus paradisi* Macf. x *Citrus tangerina* Hort)) and orange var. Salustiana (harvest June 21), treated in a drencher with PIR (1000 mg.kg⁻¹), PZ (750 mg.kg⁻¹), adjuvant (50

mg.kg⁻¹) and antifoam (200 mg.kg⁻¹) were used. This fruit was not degreened, given the later harvest date. Fruits were placed on the packing line with the following process: bath with water and NaCl₂ (200 mg.kg⁻¹), soap curtain with SOPP (10%), cascade with IMZ (1000 mg.kg⁻¹) and PZ (1500 mg.kg⁻¹) and finally, waxing, where the different treatments were conducted.

2.2.2 Application of coatings

In the first packing plant (experiment 1), the coating was applied with a swivel disc system with a flow rate of 1.5 L.ton⁻¹, used commercially by the company. The fruits were collected at the end of the drying oven (1 min and 6 s, 47-62°C). The applied treatments (selected from experimental results) were: BRIUE, CITAE, CITSUN and AC36 (1:2); and, due to their promising characteristics in recent observations and tests at citrus companies, two new coatings were also added:

- BRIUE: Brillaqua UEF6-Mercosur [18% solids (9.35% oxidized polyethylene wax and 5.7% shellac), Enzur SA Uruguay]
- CITAK: Citrosol AK UE [18% solids (Carnauba E903 and shellac), Citrosol SA Spain]

In the second packing facilities (experiment 2), the application was carried out with a spraying system with two sliding nozzles (flow rate of 0.9 L.ton⁻¹), used commercially by the company. The fruits were collected at the end of the drying oven (1 min, 43°C). The same treatments detailed in the first experiment were evaluated, and due to its promising characteristics in recent observations and tests at citrus companies, a coating was also added:

- TEC: Teycer GLK [18% solids (Carnauba and shellac), Tecnidex, Spain]

2.2.3 Evaluated physicochemical characteristics

- Initial evaluation: visual observations were made of all the fruits of each treatment with emphasis on drying and brightness, and they were weighed (initial weight). Color, firmness, juice content, SS, acidity, and ethanol and acetaldehyde concentration for each treatment were evaluated.



- **3 weeks:** net weight loss was determined, the incidence of rotten fruits, CI, and dehydration was recorded, and the general condition of the calyx was evaluated.
- **6 weeks and 6 weeks + 7 days of shelf life:** The same evaluations as in week 3 were carried out and the color, firmness, SS, acidity, and the concentration of ethanol and acetaldehyde were also evaluated for each treatment.

The mentioned variables were determined in the same way as described for the test under experimental conditions. Storage conditions were also the same.

2.2.4 Experimental design and statistical analysis

A completely randomized design was used with five repetitions per treatment. The experimental unit was 20 fruits in all cases, except for New Hall, which was 15. To evaluate color, firmness, juice percentage, SS and acidity, three random samples of 10 fruits each were taken, by evaluation date. Data was transformed if necessary (square root) for variance analysis. Mean comparison was performed using the DGC test ($p \leq 0.05$)⁽²⁵⁾.

3. Results

3.1 Evaluations at experimental conditions (mini packing line)

The following table details the external and internal quality variables of the different varieties studied.

Table 1. Peel color index (PCI), juice content (%), soluble solids (SS), acidity (%) and maturity index (MI) for each variety studied at harvest, after degreening

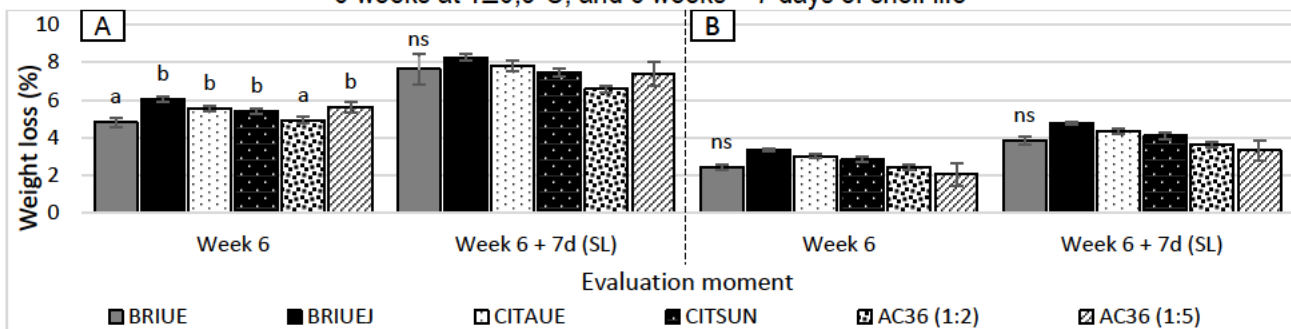
Variety	PCI	Juice (%)	SS (° Brix)	Acidity (%)	MI
Nules Clementine	2.8	41.5	11.1	1.0	11.3
Navelina	3.2	41.1	11.1	0.8	13.9

3.1.1 Weight and firmness loss

At the end of storage, after shelf life, the weight was reduced by 7.5% in Clementine and 4% in Navelina, with no differences between treatments (Figure 1). The initial average deformation of fruits after

applying the treatments was 3.17 mm in Clementine, remaining unchanged throughout storage. In Navelina, this deformation increased from an average of 1.7 to 2.5 mm, determining a significant loss of firmness, without differences between treatments (Figure 9, in supplementary material).

Figure 1. Weight loss (%) in Nules Clementine mandarin (A) and Navelina orange (B) for each treatment after 6 weeks at $1 \pm 0.5^\circ\text{C}$, and 6 weeks + 7 days of shelf life



Means (\pm standard error) followed by the same letter between treatment within each moment and variety do not differ significantly (DGC $p \leq 0.05$). References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5.



3.1.2 External and internal quality

The coatings CITAUE, CITSUN and AC36 (1:2), presented a more intense brightness at the end of the oven. BRIUEJ exhibited a more intense brightness compared to BRIUE (visual observations). However, there were no differences between treatments during refrigerated storage and subsequent shelf life.

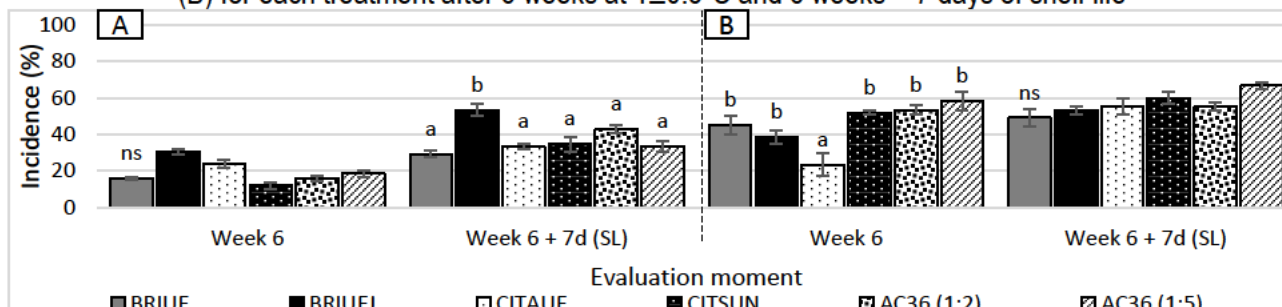
The PCI in Clementine increased to an average 3.1 after 6 weeks and to 3.9 after shelf life, with no differences between treatments. In Navelina, the PCI did not vary after 6 weeks and was 2.9 on average after shelf life, with no differences between treatments (Figure 10, in supplementary material). After shelf life, the SS remained stable, in general, increasing from 11.1 to 11.9 °Brix in Clementines

with CITAUE and AC36 (1:2 and 1:5), and from 11.1 to 11.6 °Brix in Navelina with BRIUEJ and AC36 (1:2) (Table 5, in supplementary material). The acidity decreased slightly during storage, although this decrease was not significant (Table 6, supplementary material).

3.1.3 External appearance evaluation

No symptoms of CI were observed and there was no incidence of rotten fruits during the entire storage. After shelf life, the incidence of fruits with dehydration symptoms was higher for BRIUEJ in Clementine, and no differences were observed between treatments in Navelina, although at the end of cold storage CITAUE showed less dehydration in this variety (Figure 2).

Figure 2. Incidence of dehydrated fruits (%) in mandarin var. Nules Clementine (A) and orange var. Navelina (B) for each treatment after 6 weeks at 1±0.5°C and 6 weeks + 7 days of shelf life



Means (±standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p \leq 0.05$). Referencias: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5.

At 6 weeks, 21% and 11% of the fruits presented green calyxes, while 63% and 51% showed senescent calyxes, in Clementine and Navelina, respectively. After shelf life, Clementine fruits with green calyxes decreased to 14%, while a considerable increase in the number of fruits with black calyxes (57%), was observed. Senescence was more severe in Navelina, with only 2.3% of the fruits presenting green calyxes, while 76% had black calyxes (Figure 11, in supplementary material).

3.1.4 Acetaldehyde and ethanol

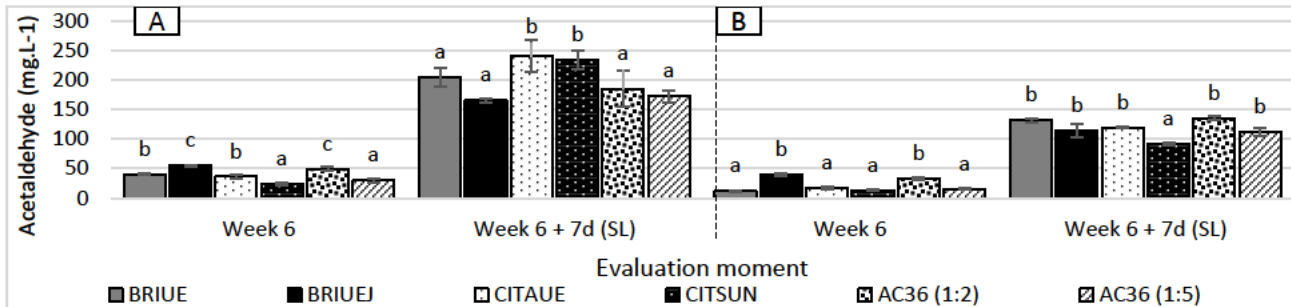
The acetaldehyde concentration slowly increased during the 6 weeks of refrigerated storage (data not

shown). The most significant increase was observed during shelf life at room temperature, and CITAUE and CITSUN showed a higher level of this compound in Clementine, while in Navelina CITSUN stood out with lower concentration (Figure 3).

The ethanol concentration showed similar behavior, highlighting the significant increase after the shelf life. BRIUEJ stands out with a higher level in both varieties after 6 weeks in cold, remaining high after the shelf life. The treatments AC36 (1:2 and 1:5) in Clementine, and BRIUE, CITAUE and AC36 (1:2 and 1:5) in Navelina, with lower levels of this compound (Figure 4).

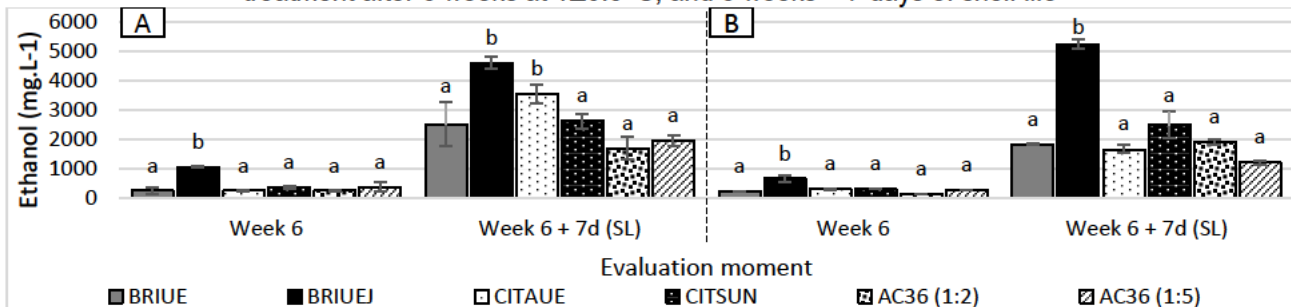


Figure 3. Acetaldehyde concentration (mg.L⁻¹) in Nules Clementine mandarin (A) and Navelina orange (B) for each treatment after 6 weeks at 1±0.5°C, and 6 weeks + 7 days of shelf life



Means (±standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC p≤0.05). References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5

Figure 4. Ethanol concentration (mg.L⁻¹) in Nules Clementine mandarin (A) and Navelina orange (B) for each treatment after 6 weeks at 1±0.5°C, and 6 weeks + 7 days of shelf life



Means (±standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC p≤0.05). References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5.

3.2 Commercial trials (packing facilities)

The following table details the external and internal quality variables of the different varieties studied at harvest. Clementine and New Hall were degreened,

given their earlier harvest and the need to improve color, which was not necessary in Nova and Salustiana, both with a later harvest date.

Table 2. Peel color index (pci), juice content (%), soluble solids (°Brix), acidity (%) and maturity index (mi) for each variety studied at harvest

Trial	Variety	Degreening	PCI	Juice (%)	SS (°Brix)	Acidity (%)	MI
1	Nules Clementine	Yes	5.0	50.0	11.6	0.9	12.4
	Navel New Hall	Yes	1.2	38.9	11.3	1.2	9.3
2	Nova	No	14.9	41.5	10.7	0.6	17.8
	Salustiana	No	6.2	53.1	12.0	0.7	16.1

Note: Degreened varieties were evaluated after this process.

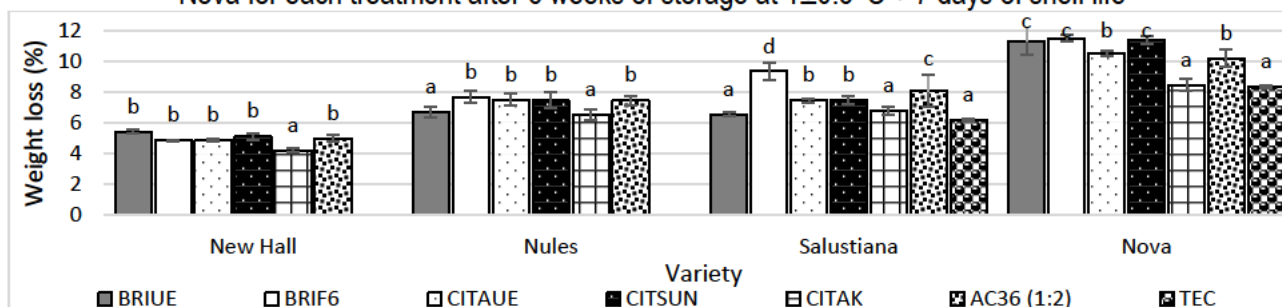


3.2.1 Weight loss and firmness

On average, weight loss was lower for orange (6.26%) than for mandarin (8.86%), in both experiments. CITAK and TCE were the treatments with the

lowest weight loss (0.7% less than the average), also BRIUE in Nules Clementine and Salustiana, presenting a 0.9% less loss than the average (Figure 5).

Figure 5. Weight loss of orange fruits var. New Hall and Salustiana, and mandarins var. Nules Clementine and Nova for each treatment after 6 weeks of storage at $1 \pm 0.5^\circ\text{C}$ + 7 days of shelf life



Means (\pm standard error) followed by the same letter between treatments within each variety do not differ significantly (DGC $p \leq 0.05$).
References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 UE dilution 1:5. Teycer GLK

A general firmness reduction was observed (greater deformation of the fruits in mm) after refrigerated storage and shelf life (Table 3, Figure 9 in supplementary material). At this moment, fruits with AC36 (1:2) showed less firmness in Clementine (deformation of 3.6 vs 3.2 mm) and New Hall (deformation

of 1.8 vs 1.6 mm), but greater in Nova (deformation of 2.0 vs 2.3 and 2.7 mm) and Salustiana (deformation of 2.7 vs 3.0 mm (Table 3)). No dehydration symptoms were observed associated with these firmness changes.

Table 3. Deformation of the fruits (mm) as an indicator of firmness for each treatment in the different varieties evaluated, at harvest and after 6 weeks of storage at $1 \pm 0.5^\circ\text{C}$ + 7 days of shelf life

Variety	Treatments	Fruit deformation (mm)	
		Harvest	Shelf life
Clementine	Average others	2.3 ns	3.2 a
	AC36 (1:2)	2.3	3.6 b
New Hall	Average others	1.3 ns	1.6 a
	CITSUN and AC36 (1:2)	1.3	1.8 b
Nova	AC36 (1:2)	1.5 ns	2.0 a
	CITAE, CITSUN, CITAK, TEC	1.5	2.3 b
	BRIUE and BRIF6	1.5	2.7 c
Salustiana	AC36 (1:2) and CITAK	1.5 ns	2.7 a
	Average others	1.5	3 b

Note: the more deformation of the fruit the less firmness.

Means (\pm standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p \leq 0.05$). References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 UE dilution 1:5. Teycer GLK.



3.2.2 External and internal fruit quality

After the application of the coatings, at the exit of the oven, BRIF6, CITAK and TEC waxes stood out for their more intense shine, while AC36 wax (1:2) gave a smoother and more natural shine to the fruit. Regarding the drying, AC36 (1:2) and CITAK coatings showed a faster drying, however, at the end of the line (after the disposal and packing stages) all treatment fruits were dry. After cold storage, brightness differences between treatments decreased (visual observations).

The PCI remained stable throughout the storage in the four varieties, with some exceptions: after shelf life, it fell from 8.7 to 7.6 in Nova with BRIUE, BRIF6, CITAUE, CITSUN and AC36 (1:2) treatments, and from 4.1 to 3.7 in Salustiana with AC36 (1:2), CITAUE and CITSUN treatments (Figure 10, in supplementary material).

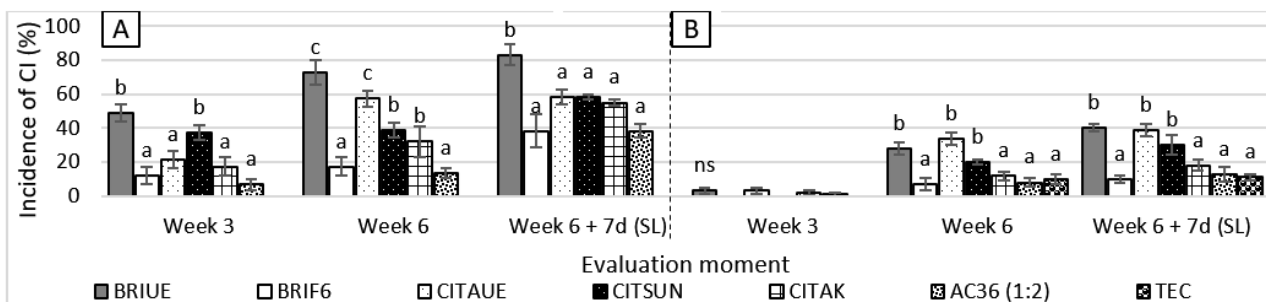
Soluble solids varied in New Hall, decreasing from 11.3 to 10.0 °Brix with BRIUE, and increasing to 13.6 °Brix with CITAK and CITAUE. In Clementine it decreased from 11.9 to 11.0 °Brix with AC36 (1:2),

while in Nova it increased from 10.7 to 11.5 °Brix with BRIUE and BRIF6 (Table 5). Acidity slightly decreased in the 4 varieties, although this decrease was not significant (Table 6).

3.2.3 External appearance evaluation

No symptoms of CI were observed in Clementine, while in Nova a slight incidence was detected after 6 weeks of storage, ascending to 7.3% of the fruits after shelf life and without differences between treatments. In oranges, CI was more severe, with clear differences between treatments. In New Hall, symptoms were observed after 3 weeks, with a higher incidence for BRIUE and CITSUN (43.0%), compared to the rest of the treatments (18.9%). After shelf life, the intensity of CI increased and the injuries became more visible, differing only BRIUE (83.2%) from the rest of the treatments (49.7%). In Salustiana, CI was observed in all treatments after 6 weeks and was higher on average for BRIUE, CITAUE and CITSUN (27.3%) compared to the rest of the treatments (9.3%). Incidence increased slightly during shelf life, maintaining the same differences between coatings (36.3 vs 13.0%, Figure 6).

Figure 6. Incidence of chilling injury (CI) on orange fruits var. New Hall (A) and Salustiana (B) for each treatment after 3 and 6 weeks of storage at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life



Means (\pm standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p \leq 0.05$). References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 UE dilution 1:5. Teycer GLK.

The calyx senescence increased during storage and was aggravated after shelf life. It was higher in degreened varieties, especially in New Hall. No differences were observed between the coatings in

New Hall and Nova, while Clementines with BRIUE, and Salustianas with BRIF6, CITAUE, CITAK and TEC presented a higher percentage of green calyxes (Table 4, Figure 11 in supplementary material).



Table 4. General calyx condition (%) in the four evaluated varieties according to treatments, after 6 weeks of storage at $1\pm 0.5^{\circ}\text{C}$ + 7 days of shelf life

Variety	Treatments	Green	Senescent	Black	Fallen
Clementine	BRIUE	47.0	36.0	17.0	0.0
	Average others	30.2	35.0	32.8	2.0
New Hall	Average all	0.0	0.0	83.6	16.4
Nova	Average all	96.9	3.1	0.0	0.0
Salustiana	BRIF6 – CITAUE – CITAK - TEC	92.5	7.5	0.0	0.0
	BRIUE - AC36 (1:2) - CITSUN	81.7	18.3	0.0	0.0

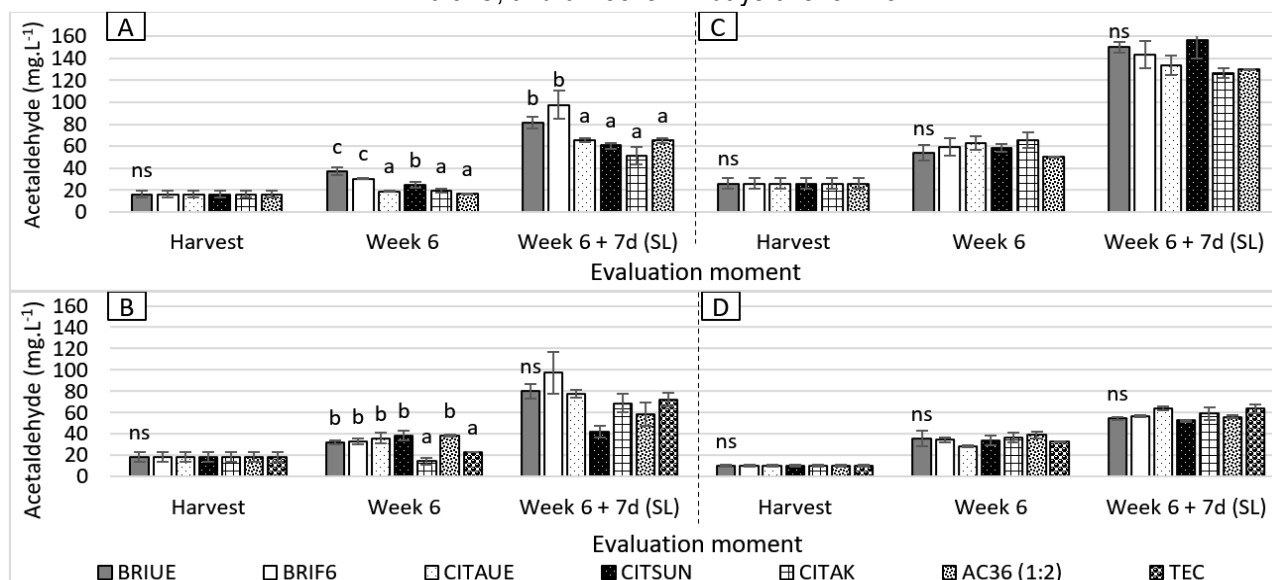
Note: For each variety, the treatments were grouped according to the differences by statistical analysis (DGC $p\leq 0.05$).
References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 dilution 1:2; TEC: Teycer GLK.

3.2.4 Acetaldehyde and ethanol

A slight increase in the acetaldehyde concentration was observed during the refrigerated storage which was accentuated significantly after shelf life. In Clementine, it increased from 16.2 to 60.8 $\text{mg}\cdot\text{L}^{-1}$ on average, while the treatments BRIUE and BRIF6 stood

out with an even greater increase (89.7 $\text{mg}\cdot\text{L}^{-1}$ on average). In the rest of the varieties, no differences were observed between treatments, being the increases, on average, in Nova from 18.3 to 71.1 $\text{mg}\cdot\text{L}^{-1}$, in New Hall from 26.0 to 140.0 $\text{mg}\cdot\text{L}^{-1}$ and in Salustiana from 10.3 to 58.2 $\text{mg}\cdot\text{L}^{-1}$ (Figure 7).

Figure 7. Acetaldehyde concentration ($\text{mg}\cdot\text{L}^{-1}$) in mandarins var. Nules Clementine (A) and Nova (B), and in oranges var. New Hall (C) and Salustiana (D), for each treatment at harvest, after 3 and 6 weeks of storage at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life



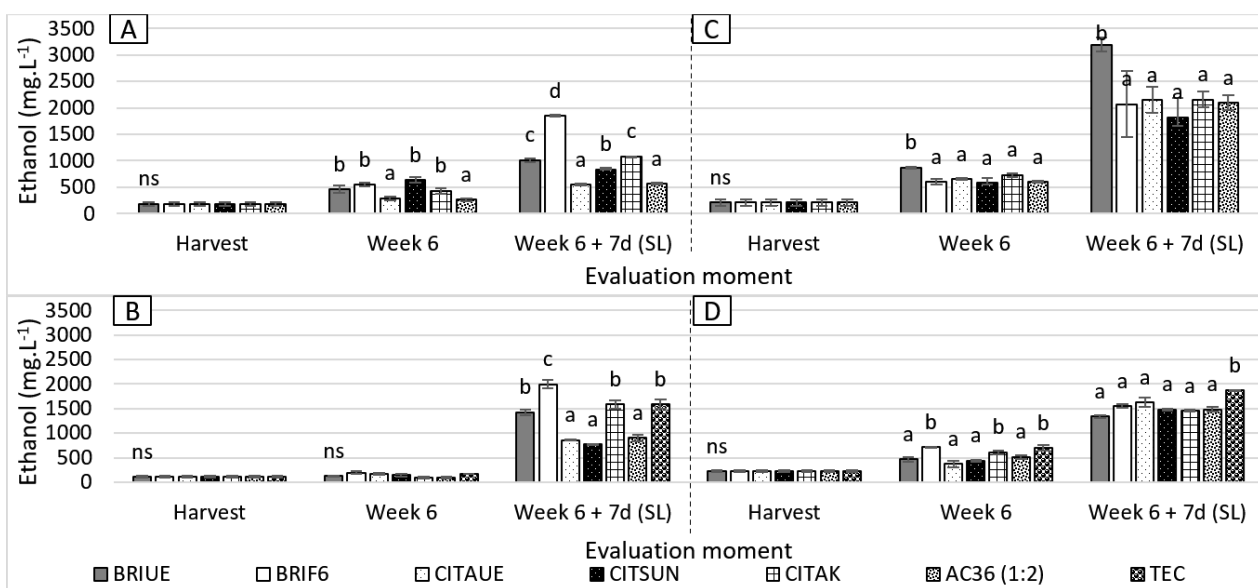
Means (\pm standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p\leq 0.05$). References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 UE dilution 1:5; Teycer GLK.



The ethanol concentration showed a similar trend to that of acetaldehyde and increased mainly after shelf life. In Clementine, it increased from 183.3 to 977.6 mg.L⁻¹, with the treatment BRIF6 having the highest concentration (1854.2 mg.L⁻¹) and the treatments AC36 (1:2) and CITAUE with lower concentration (559.5 mg.L⁻¹ on average). In Nova, it increased from 110.1 to 1532.6 mg.L⁻¹, being BRIF6 the

treatment with the highest concentration (1997.0 mg.L⁻¹), and the treatments CITAUE, CITSUN and AC36 (1:2) with lower concentration (843.3 mg.L⁻¹). In New Hall, it increased from 213.4 to 2059.9 mg.L⁻¹ on average, with the exception of BRIUE (3196.2 mg.L⁻¹). In Salustiana, the increase was from 225.6 to 1490.2 mg.L⁻¹ on average, TEC standing out with the highest concentration (1873.7 mg.L⁻¹, Figure 8)

Figure 8. Ethanol concentration (mg.L⁻¹) in Nules Clementine (A) and Nova (B) mandarins, and in New Hall (C) and Salustiana (D) oranges for each treatment at harvest, after 6 weeks of storage at 1 ± 0.5°C, and 6 weeks + 7 days of shelf life



Means (±standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC p ≤ 0.05). References: BRIUE: Brillaqua UE; BRIF6: Brillaqua UEF6; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; CITAK: Citrosol AKUE; AC36 (1:2): Frutcoat AC-36 UE dilution 1:5. Teycer GLK

4. Discussion

The environmental conditions during the citrus fruit packing in Uruguay (high relative humidity and medium-low temperatures), as well as the diversity of markets at different distances and with varied requirements (quarantine treatments, external and internal quality requirements), lead to the selection of suitable coatings. The evaluation of new coatings as alternatives to the BRIUE control, widely used by all Uruguayan citrus companies, showed interesting results and supported the decision to change at a commercial level.

In the experimental evaluation, CITAUE and CITSUN showed higher brightness levels and good general appearance. In the commercial evaluation, BRIF6, CITAK and TEC coatings stood out. The increased concentration of shellac in BRIF6 compared to BRIUE, as well as the presence of carnauba in the composition of CITAK and TEC are relevant factors that contribute to superior visual quality and a more intense brightness. Carnauba is the compound that generates the highest shine, followed by shellac and finally oxidized polyethylene⁽²¹⁾⁽²²⁾. Differences between coatings with the same ingredients could be due to the concentration of each particular compound and the other chemical components that



accompany the formulation, such as surfactants and emulsifiers⁽¹⁾⁽²²⁾. The AC36 (1:2) coating gave the fruit a natural appearance in terms of brightness. Diluting this coating to 1:5 in the experimental phase, negatively influenced the general appearance of the fruits, presenting a lower brightness. According to Hassan and others⁽⁵⁾, a very thin layer of wax, or with a very low percentage of solids, may not be effective in preventing senescence and fruit quality loss. As the storage period advanced, some authors reported decreases in fruit brightness with coatings based on oxidized polyethylene⁽⁷⁾, or oxidized polyethylene and shellac⁽¹⁵⁾. During the commercial evaluation, the same trend was observed in the three most promising coatings in terms of brightness, without detecting large differences with the rest of the treatments after shelf life.

Fruit weight loss increased during storage, in general, it was greater in mandarin than in orange. This may be due to anatomical differences⁽⁵⁾, such as fruit size; since smaller fruits have greater interaction with the outside, or to the peel thickness, which is greater in oranges (data not shown). These authors also describe that the weight loss of fruits is due to the respiratory process, the loss of humidity and some oxidation processes, therefore, the conservation temperature and the high relative humidity were key factors in the prevention of weight loss. Differences between treatments in the experimental evaluation were not observed, nevertheless, in the commercial evaluation, treatments with carnauba (CITAK and TEC) showed a smaller weight loss (5.7% in oranges and 7.8% in mandarins) compared to the rest (6.4% in oranges, 9.2% in mandarins). Carnauba is the ingredient/compound that most limits gaseous interchange, therefore, it reduces the weight loss caused mainly by dehydration. In this aspect, it is followed by shellac and, finally, by oxidized polyethylene⁽²²⁾. Therefore, the coating composition is a factor that impacted directly on the weight loss.

Firmness loss was more intense in oranges than in mandarins, especially in Navelina and New Hall. This was directly associated with weight loss, which was greater in these varieties. In general, no differences were observed in the commercial evaluation between coatings, except for AC36 (1:2) which

further reduced firmness in New Hall and Clementine but was the firmest in Nova and Salustiana.

The evaluated coatings did not impact the color of the fruits since the PCI remained unchanged during refrigerated storage for most treatments and varieties. Variations in the color of fruits coated with oxidized polyethylene waxes have been observed, but not with shellac coatings, although storage temperature was higher: 2-3°C and 9-10°C⁽²¹⁾. A decrease in the PCI was observed in Nova, only in those with carnauba-free coatings, after shelf life. In Salustiana, this decrease was observed with the CITAE, CITSUN and AC36 (1:2) coatings.

In general, coatings did not affect the internal quality, with some exceptions regarding the SS. Variations depended mainly on the evaluated varieties since coatings did not show a constant between them. The reduction of acidity content caused by the use of acids as an energy source, as well as the increase in soluble solids due to the hydrolytic activity of starch, are metabolic processes that depend directly on temperature⁽⁵⁾, therefore, surely, conservation at 1°C influenced these results. Other authors did not observe differences in the SS content and acidity in fruit stored at 5°C⁽¹⁰⁾ and 7°C either⁽¹²⁾. However, there are also records of acidity reductions and SS increases after prolonged storage at 5°C, although these were accentuated after periods of shelf life at 20-25°C⁽⁵⁾⁽⁹⁾⁽¹⁵⁾⁽¹⁶⁾⁽¹⁷⁾.

Regarding the general condition of the calyx, a senescence increase was observed over time, which was aggravated after the rise in temperature, during shelf life. Degreening was a key factor that favored the blackening and senescence of the calyx, detecting differences in the varieties with or without ethylene treatment. This application generates a color change and accelerates senescence, producing premature fruit aging, an increase in perspiration with weight loss, desiccation of calyxes and even an increase in rotten fruits⁽²⁾⁽²⁶⁾⁽²⁷⁾. Within the degreened varieties, in Clementine, BRIUE limited senescence compared to the rest of the treatments (47 vs 30% of green calyxes respectively, after shelf life), while in New Hall senescence was so severe that no treatment stood out (100% black and/or fallen calyxes after shelf life). Among the varieties without degreening, in Salustiana, the most



occlusive coatings were highlighted (BRIF6, CITAK and TEC) together with CITAUE, limiting senescence compared to the rest of the evaluated coatings (93 vs 82% of green calyxes respectively, after shelf life). In Nova mandarin, despite having an advanced state of maturity, the senescence of the calyxes was minimal, due to a varietal characteristic, with no differences between treatments (97% of green calyxes after shelf life).

The application of wax coatings is described as a postharvest handling tool that can minimize the appearance or aggravation of the symptoms of chilling injury⁽²⁾⁽²⁰⁾⁽²²⁾. Results obtained demonstrate that the coatings BRIF6, CITAK, AC36 (1:2) and TEC could prevent or reduce the intensity of the symptoms of CI in New Hall and Salustiana oranges, compared to BRIUE, CITAUE and CITSUN, which had a higher incidence of CI. All these species, under these storage conditions⁽²⁾⁽¹⁹⁾, are susceptible to this disorder, determining differential behavior of the coatings in interaction with the variety under study.

Coatings reduce gas exchange between fruits and the environment, especially as the contents of carnauba and shellac are higher, and the content of oxidized polyethylene is lower⁽²²⁾⁽²⁸⁾. The lower this exchange, the lower the water loss (less dehydration and weight loss), but also the production of acetaldehyde and ethanol could be stimulated, as temperature increases and, with it, respiration. Fermentative processes can occur if the oxygen demand exceeds its capacity to enter the fruit, which would lead to the development of off-flavors and a higher acetaldehyde and ethanol concentration⁽²⁶⁾. Detecting these off-flavors in citrus fruits is associated with ethanol levels above 800-5000 mg.L⁻¹⁽⁷⁾, 900 mg.L⁻¹⁽¹¹⁾, 1500 mg.L⁻¹⁽⁸⁾⁽⁹⁾ or 2000 mg.L⁻¹⁽²⁹⁾

The evolution of the acetaldehyde and ethanol concentration registered similar behaviors in all the evaluated varieties, with a progressive increase during storage that was accentuated after shelf life. This coincides with what various researchers have observed⁽⁶⁾⁽¹⁰⁾⁽¹¹⁾⁽¹⁶⁾⁽²⁴⁾. Once again, storage temperature was influential, being one of the main factors that stimulate the synthesis of both compounds⁽²⁶⁾.

In the experimental phase, the concentration of acetaldehyde and ethanol, in general, were higher in Clementine than in Navelina. This may be due to

anatomical differences in the peel of both species, which provide different levels of permeability and gas exchange as well as due to genetic differences in the natural production of both compounds. In this sense, differences were observed when comparing W. Murcott mandarin and Star Ruby grapefruit, concluding that the grapefruit flavedo is thinner and with fewer oil glands (impermeable to gases), and the albedo is less dense than that of W. Murcott, so the diffusion of gases would be greater, which can reduce the formation of volatile compounds derived from anaerobic respiration⁽¹⁴⁾. After shelf life, no great differences were observed between treatments with respect to acetaldehyde, while BRIUEJ was the coating that showed a higher concentration of ethanol in Clementine (4619 vs 2201 mg.L⁻¹ average), and Navelina (5267 vs 1824 mg.L⁻¹ on average).

In the experiments under commercial conditions, the accumulation of both volatiles was higher in New Hall, reaching the detectable threshold of ethanol (2060 mg.L⁻¹ on average) and widely exceeding it in the case of the control (BRIUE, 3196 mg.L⁻¹). In mandarins, coatings that reduced weight loss (BRIF6, CITAK and TEC), being more occlusive, favored the synthesis of this volatile (1467 mg.L⁻¹ in Clementine and 1724 mg.L⁻¹ in Nova), while CITAUE, CITSUN and AC36 (1:2) stood out with lower concentration (652 mg.L⁻¹ on average in Clementine and 843 mg.L⁻¹ on average in Nova). In Salustiana, TEC favored, once again, ethanol formation (1874 vs 1490 mg.L⁻¹ on average in the other coatings). These differences are associated with a different composition of the waxes. Not only the main coating components are important, but also their concentration⁽²²⁾. Depending on the destination of the fruits, the composition of coatings should vary, choosing for short trips carnauba or shellac components (higher brightness) and for longer transports, oxidized polyethylene, assuming the loss of brightness⁽²²⁾.

5. Conclusions

Differences in the behavior of the evaluated coatings were observed which were related to the citrus variety. Coatings did not negatively affect the external (visual) quality, the firmness or the internal quality of the fruit compared to Brillaqua UE 18% (BRIUE-



control), so all of them could be used as an alternative, depending on each company's requirements. The Citrosol AK and Teycer GLK coatings with carnauba and shellac were the ones with the lowest weight loss after storage and shelf life.

Brightness and general appearance of the fruit were superior in the Brillaqua F6, Citrosol AK and Teycer GLK treatments at the end of the drying oven. After shelf life, no great differences were observed, highlighting Concentrol AC-36 (1:2) and Citrosol AUE.

In the most susceptible varieties to CI (New Hall and Salustiana), most of the coatings offered better protection than Brillaqua UE 18%, with the exception of Citrosol (AUE and Sunseal) in Salustiana.

The continuous control of storage temperature is very important regarding the levels of ethanol and acetaldehyde. The concentration of these volatiles depended on the type of coating, its application form and the application of the degreening treatment. In the commercial phase, in general, no coating exceeded the detection threshold of 2000 mg.L⁻¹, except for BRIUE in New Hall. The most occlusive treatments provide greater brightness and less weight loss, presenting higher concentrations of these volatiles. In these cases, it is important to achieve strict temperature control during periods of shelf life, especially in mandarins.

Finally, it is concluded that Brillaqua F6, Citrosol AK and Teycer GLK coatings were the most promising for postharvest application on citrus fruits and subsequent storage at 1°C. These presented a higher level of brightness and a superior general appearance of the fruit, less weight loss and levels of acetaldehyde and ethanol that did not exceed the proposed thresholds. It is important to bear in mind that its use on overripe fruit or with a more severe degreening process can cause the appearance of off-flavors in some varieties if the storage temperature increases.

The Concentrol AC-36 (1:2) and Citrosol AUE coatings are not ruled out, as they had acceptable levels of brightness, especially after conservation, as well as acceptable general visual appearance and levels of acetaldehyde and ethanol.

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Author contribution statement

Fernandez, G. collected the data, performed the analyses and wrote the article. Scaparoni, F., Sisquella, M., Pintos, P., Luque E., Moltini A. collected the data. Lado, J. performed the analyses and wrote the article.

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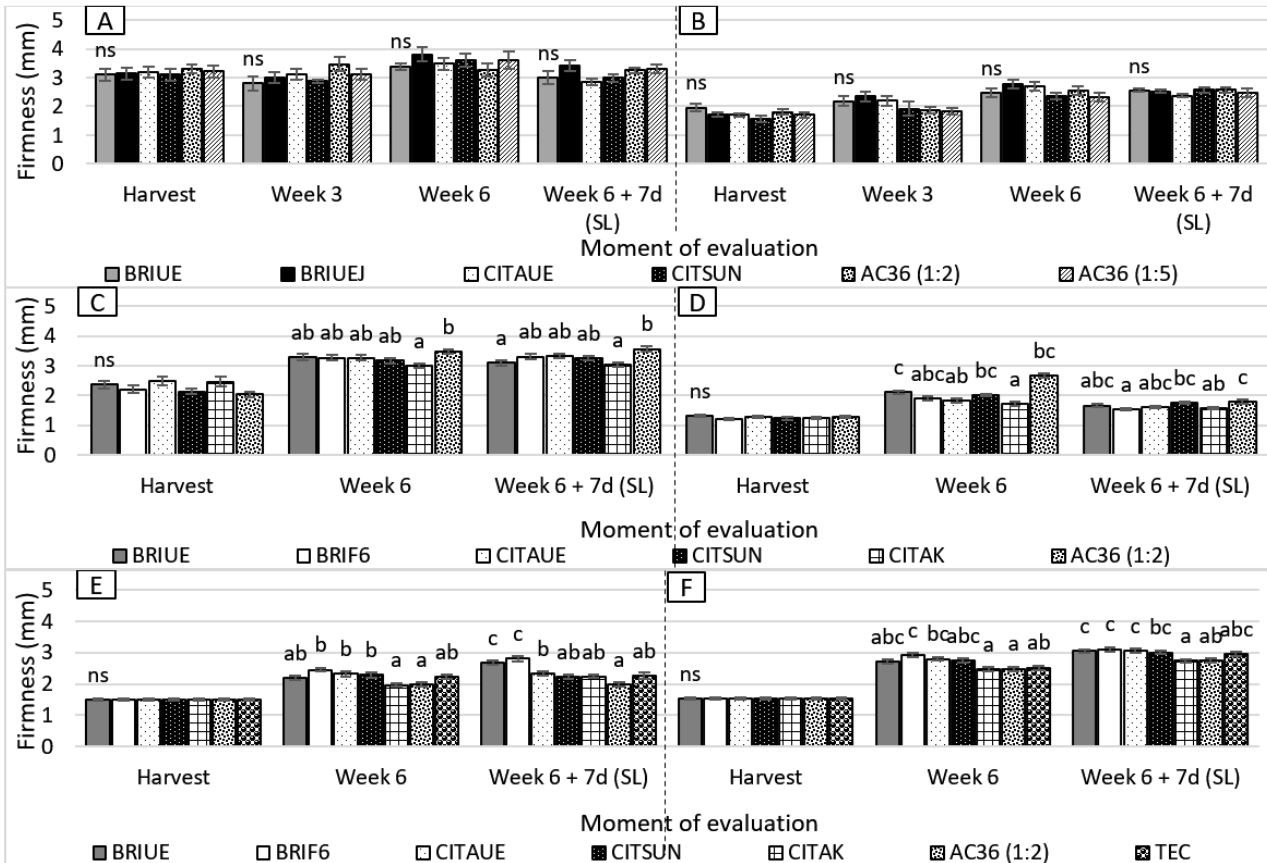
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Supplementary material

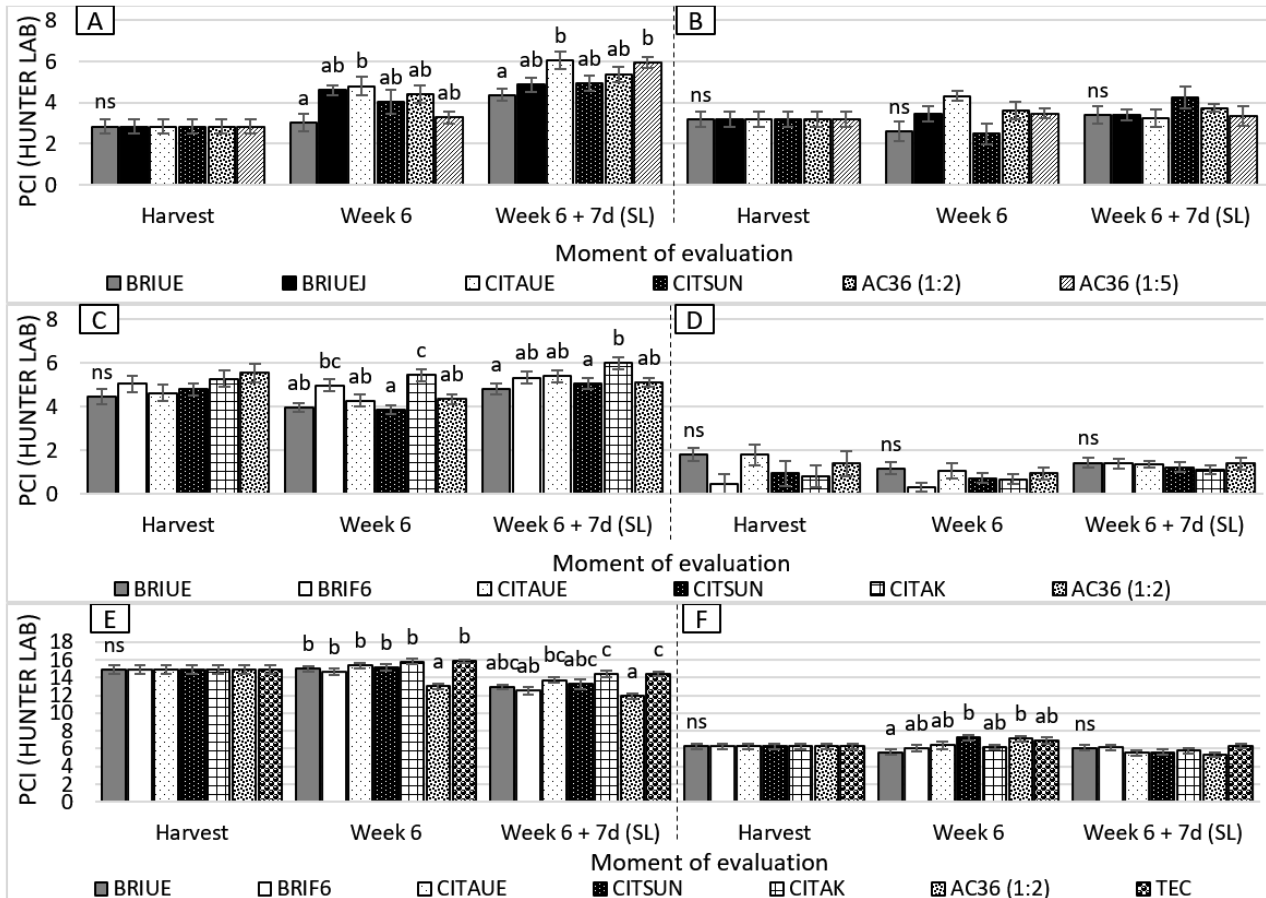
Figure 9. Firmness (expressed as mm of fruit deformation) in Nules Clementine mandarin (A) and Navelina orange (B) from the experimental evaluation, and in Nules Clementine mandarin (C), New Hall orange (D), Nova mandarin (E) and Salustiana orange (F) from the commercial evaluation, for each treatment at harvest and after 3 and 6 weeks at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life



Means (\pm standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p \leq 0.05$). References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5; BRIF6: Brillaqua UEF6; CITAK: Citrosol AKUE; TEC: Teycer GLK.



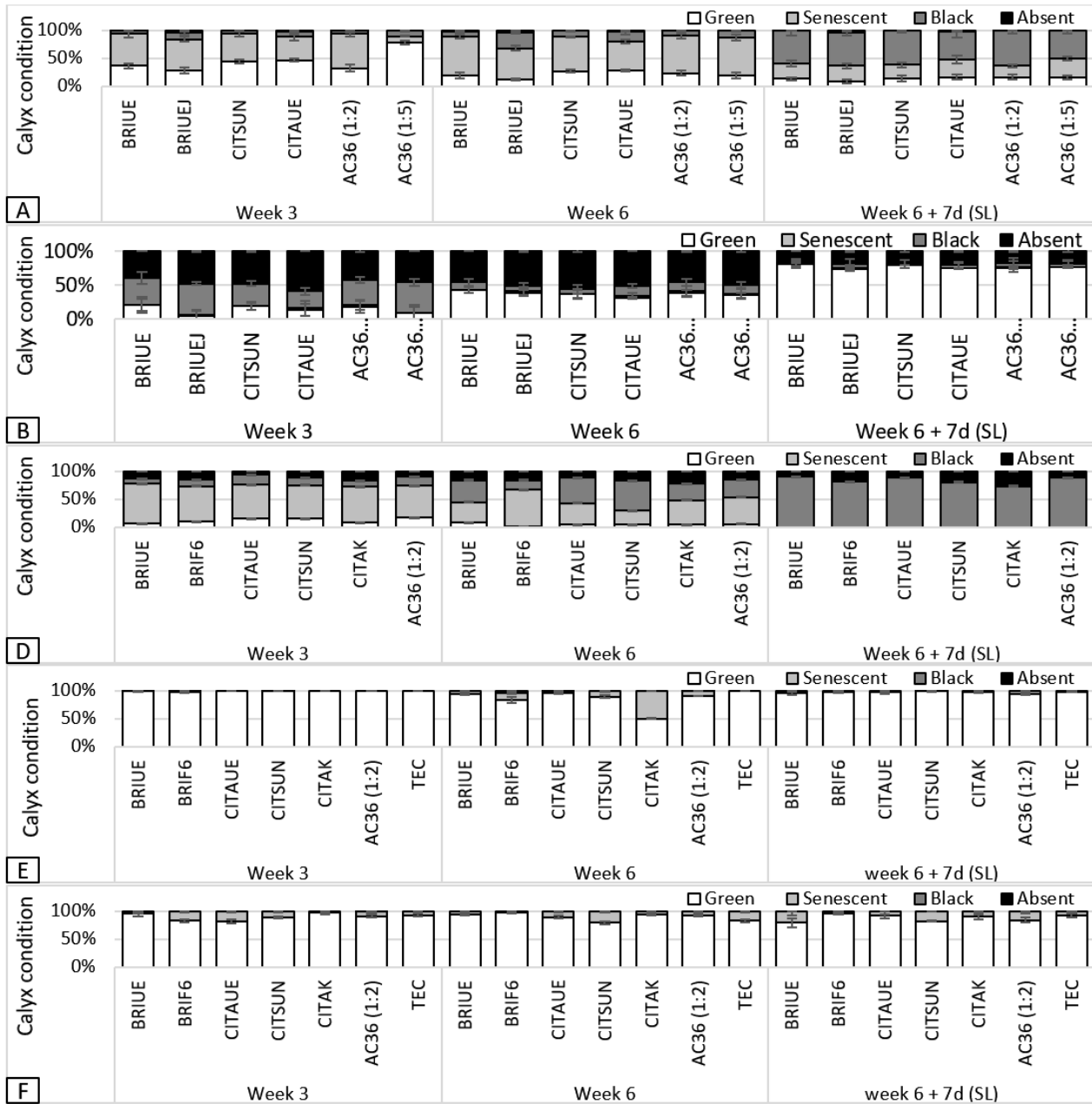
Figure 10. Peel color index (PCI, CIELAB) in Nules Clementine mandarin (A) and Navelina orange (B) from the experimental evaluation, and in Nules Clementine mandarin (C), New Hall orange (D), Nova mandarin (E) and Salustiana orange (F) from the commercial evaluation, for each treatment at harvest and after 3 and 6 weeks at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life



Means (\pm standard error) followed by the same letter between treatments within each moment and variety do not differ significantly (DGC $p \leq 0.05$). References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5; BRIF6: Brillaqua UEF6; CITAK: Citrosol AKUE; TEC: Teycer GLK.



Figure 11. General calyx condition, categorized according to its senescence as green, senescent, black or absent, in Nules Clementine mandarin (A) and Navelina orange (B) from the experimental evaluation, and in Nules Clementine mandarin (C), New Hall orange (D), Nova mandarin (E) and Salustiana orange (F) from the commercial evaluation, for each treatment, after 3 and 6 weeks at 1 ± 0.5°C, and 6 weeks + 7 days of shelf life



References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilución 1:2; AC36 (1:5): Frutcoat AC-36 UE dilución 1:5; BRIF6: Brillaqua UEF6; CITAK: Citrosol AKUE; TEC: Teycer GLK.



Table 5. Soluble solids content (°Brix) in Nules Clementine mandarin and Navelina orange from the experimental evaluation, and in Nules Clementine mandarin, New Hall orange, Nova mandarin and Salustiana orange from the commercial evaluation, for each treatment at harvest and after 3 and 6 weeks at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life

Variety	Treatment	Harvest	Week 3	Week 6	Week 6 + 7d SL
Nules Clementine	BRIUE	11.1	10.5	10.6	10.8
	BRIUEJ	11.1	11.3	11.1	10.9
	CITAUE	11.1	11.7	11.6	11.8
	CITSUN	11.1	11.7	10.7	10.8
	AC36 (1:2)	11.1	11.3	11.1	11.6
	AC36 (1:5)	11.1	11.7	11.0	12.1
Navelina	BRIUE	11.1	11.1	10.5	11.5
	BRIUEJ	11.1	11.2	11.3	11.6
	CITAUE	11.1	11.1	11.5	10.7
	CITSUN	11.1	12.4	11.0	11.3
	AC36 (1:2)	11.1	11.5	11.8	11.6
	AC36 (1:5)	11.1	11.3	11.6	10.6
Nules Clementine	BRIUE	11.6		11.6	11.5
	BRIF6	11.4		11.5	11.2
	CITAUE	11.4		11.3	11.5
	CITSUN	11.6		11.7	11.7
	CITAK	11.7		11.6	11.5
	AC36 (1:2)	11.9		11.3	11.0
New Hall	BRIUE	13.7		10.2	10.0
	BRIF6	10.7		10.4	10.0
	CITAUE	10.9		10.4	13.7
	CITSUN	10.9		10.4	10.4
	CITAK	10.7		10.0	13.5
	AC36 (1:2)	11.1		10.3	10.3
Nova	BRIUE	10.7		11.3	11.5
	BRIF6	10.7		11.1	11.4
	CITAUE	10.7		11.4	11.0
	CITSUN	10.7		11.5	10.8
	CITAK	10.7		10.7	10.5
	AC36 (1:2)	10.7		10.8	11.0
	TEC	10.7		11.4	10.7
Salustiana	BRIUE	12.0		11.8	12.0
	BRIF6	12.0		11.7	12.0
	CITAUE	12.0		11.7	12.1
	CITSUN	12.0		12.2	11.9
	CITAK	12.0		11.7	12.1
	AC36 (1:2)	12.0		12.0	11.9
	TEC	12.0		11.7	12.0

References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5; BRIF6: Brillaqua UEF6; CITAK: Citrosol AKUE; TEC: Teycer



Table 6. Acidity content (%) in Nules Clementine mandarin and Navelina orange from the experimental evaluation, and in Nules Clementine mandarin, New Hall orange, Nova mandarin and Salustiana orange from the commercial evaluation, for each treatment, at harvest and after 3 and 6 weeks at $1\pm 0.5^{\circ}\text{C}$, and 6 weeks + 7 days of shelf life

Variety	Treatment	Harvest	Week 3	Week 6	Week 6 + 7d SL
Nules Clementine	BRIUE	1.0	0.8	0.7	0.7
	BRIUEJ	1.0	0.7	0.7	0.8
	CITAUE	1.0	0.8	0.7	0.7
	CITSUN	1.0	0.8	0.7	0.8
	AC36 (1:2)	1.0	0.8	0.7	0.7
	AC36 (1:5)	1.0	0.8	0.8	0.7
Navelina	BRIUE	0.8	0.7	0.6	0.7
	BRIUEJ	0.8	0.7	0.7	0.7
	CITAUE	0.8	0.7	0.7	0.7
	CITSUN	0.8	0.6	0.6	0.6
	AC36 (1:2)	0.8	0.8	0.6	0.6
	AC36 (1:5)	0.8	0.7	0.5	0.6
Nules Clementine	BRIUE	0.9		0.9	0.8
	BRIF6	0.9		0.9	0.9
	CITAUE	0.9		0.9	0.8
	CITSUN	0.9		0.9	0.9
	CITAK	0.9		0.9	0.9
	AC36 (1:2)	1.0		0.8	0.8
New Hall	BRIUE	1.1		1.1	1.1
	BRIF6	1.3		1.1	1.0
	CITAUE	1.3		1.2	1.1
	CITSUN	1.2		1.1	1.1
	CITAK	1.1		1.3	1.0
	AC36 (1:2)	1.3		1.1	1.1
Nova	BRIUE	0.6		0.5	0.5
	BRIF6	0.6		0.5	0.6
	CITAUE	0.6		0.6	0.5
	CITSUN	0.6		0.5	0.5
	CITAK	0.6		0.5	0.5
	AC36 (1:2)	0.6		0.4	0.4
	TEC	0.6		0.6	0.4
Salustiana	BRIUE	0.8		0.7	0.7
	BRIF6	0.8		0.6	0.7
	CITAUE	0.8		0.7	0.8
	CITSUN	0.8		0.6	0.7
	CITAK	0.8		0.7	0.6
	AC36 (1:2)	0.8		0.6	0.8
	TEC	0.8		0.6	0.7

References: BRIUE: Brillaqua UE; BRIUEJ: Brillaqua UEJ; CITAUE: Citrosol AUE; CITSUN: Citrosol Sunseal; AC36 (1:2): Frutcoat AC-36 UE dilution 1:2; AC36 (1:5): Frutcoat AC-36 UE dilution 1:5. Brillaqua UEF6; CITAK: Citrosol AKUE; TEC: Teycer GLK.