# Chemical characterization of Valencia, Salustiana and Lane Late sweet oranges fruits in Frederico Westphalen-RS

Paulo de Tarso Lima Teixeira<sup>1</sup>, Sandro Rogério Giacomelli<sup>1</sup> & Mateus Ogliari<sup>1</sup>

# SUMMARY

The chemical characterization of peel and pulp of Valencia Lane Late and Salustiana sweet oranges [*Citrus sinensis* (L.) Osbeck], grafted on *Poncirus trifoliata* (L.) Raf. grown in Polo citrus collection of Technological Modernization of Medium High Uruguay. The experiment was conducted under geographic coordinates  $27^{\circ}22$  'S;  $53^{\circ}25$ ' W, 480 m above sea level, the city of Frederico Westphalen - RS. The climate, according to Köppen classification is Cfa, tropical without dry season and temperature of the warmest month greater than  $22^{\circ}$  C. It was found that the juice of the show a considerable content of ascorbic acid, to 40.71 mg.100 mL<sup>-1</sup>, at Salustiana sweet orange. Moreover, they are significant sources of polyphenols and also have high antioxidant activity, with higher values in the pulp. The Salustiana cultivar reached the highest polyphenol content, 51.1%. However, it was found that none of the other cultivars showed significant levels of carotenoids, reaching levels of up to 0.64 g mg.100<sup>-1</sup> in the albedo of the Salustiana. Thus, it can be concluded that the residues of other species have chemical characteristics that enable their reuse as a source of polyphenols as well as an antioxidant.

**Index terms:** citrus, sweet orange, maturity index, ascorbic acid, carotenoids, polyphenols, antioxidant activity.

# Caracterização química dos frutos das laranjeiras doces Valência, Salustiana e Lane Late em Frederico Westphalen-RS

## **RESUMO**

A caracterização química de cascas e polpas dos frutos das laranjeiras doces Valência, Salustiana e Lane Late [*Citrus sinensis* (L.) Osbeck], enxertadas sobre *Poncirus trifoliata* (L.) Raf. cultivadas na coleção de citros do Polo de Modernização Tecnológica do Médio Alto Uruguai. O experimento foi conduzido sob as coordenadas geográficas 27°22 'S; 53°25' W, a 480 m acima do nível do mar na cidade de Frederico Westphalen-RS. O clima da região, conforme classificação de Köppen, é do tipo Cfa, subtropical, sem estação seca e temperatura do mês mais quente maior que 22°C. Através dos resultados obtidos constatou-se que o suco das espécies estudadas apresentam um teor considerável de ácido ascórbico, de até 40,71 mg.100 mL<sup>-1</sup>, no caso da laranja Salustiana. Além disso, são consideráveis fontes de polifenois e também apresentam elevada atividade antioxidante, apresentando maiores valores na polpa. A cultivar Salustiana atingiu o maior teor de polifenois, 67,79%. No entanto, foi possível constatar que nenhuma das cultivares estudadas

<sup>&</sup>lt;sup>1</sup> Universidade Regional Integrada do Alto Uruguai e das Missões - URI, Itapagé, RS, Brazil

**Corresponding author:** Paulo de Tarso Lima Teixeira. Universidade Regional Integrada do Alto Uruguai e das Missões – URI, Rua Assis Brasil, nº 709, Câmpus Frederico Westphalen, CEP 98400-000, Itapagé, RS, Brazil. E-mail: teixeira@uri.edu.br

apresentaram teores significativos de carotenoides, chegando a teores de no máximo 0,64 mg.100 g<sup>-1</sup> no albedo da cultivar Salustiana. Assim, pode-se concluir que os resíduos das espécies estudadas apresentam características químicas que viabilizam sua reutilização como fonte de polifenois e também como antioxidante.

**Termos de indexação:** citrus, laranja doce, índice de maturação, ácido ascórbico, carotenoides, polifenois, atividade antioxidante.

#### **INTRODUCTION**

Citrus fruits are grown in various regions of the world and Brazil and generate around R\$ 9 billion and 250 thousand jobs at the Brazil. Orange is the flagship in particular in relation to the world market for orange juice. The Brazilian production of estimated for 2016 by EMATER/RS was 18.4 million tonnes in an area of 715 thousand hectares. Or even 666,340 hectares with production of 15,674,994 tons and average productivity of 23.5 tonnes per hectare (IBGE/PAM, 2016, cited by Kist et al., 2016). Sailing today orange is the fruit more produced in the country.

The orange juice is the product of weight in national, being the most exported as concentrated juice. Brazil has contributed with 31% of the orange produced for juice and 55% of world production and 73% of the export of juice in the world scenario. The largest buyer of Brazilian juice is the European Union, followed by the United States of America and Canada (Kist et al., 2016).

In Rio Grande do Sul State, citriculture is predominantly in small properties with family labor and has an enormous potential to be explored due to regions with climatic conditions favorable to the production of fruits with different quality (Oliveira & Scivittaro, 2011; Koller, 2006). Most of the production of oranges has the objective of supplying the juice with a predominance of the cultivation of Valencia orange and to a lesser extent of navel oranges, the latter only intended for *in natura* consumption (Oliveira & Scivittaro, 2011).

The wastes arising from citrus cultivation have a significant commercial value that makes their reuse, since they present in their composition antioxidant compounds, fiber, vitamins, among others, can be used in aromatic essences obtained in the concentration of the juice; R-limonene used in the manufacture of paints and solvents; soybean meal of citrus pulp for the production of feed; orange pulp used by food and beverage industries; and, the essential oils of the shell used as inputs in the food industry, beverages, cosmetics and perfumes (Pereira & Cardoso, 2012). A large amount of livestock waste is generated by the processing of citrus, between 30 and 40% of waste

are not used. It is interesting both from environmental and economically, adding value to them, using for this scientific and technological research in order to provide an efficient use, economical and safe (Pereira & Cardoso, 2012).

In the case of citric fruits, consider themselves as quality factors: size, weight, thickness of the shell, color, texture, content of juice, total soluble solids, total acidity, soluble solids/acidity (*ratio*), nutrition, carotenoid content and existence of chemical residues of agricultural pesticides (Borges & Pio, 2003).

The sensory attributes, such as aroma, flavor, texture and color, are influenced significantly by the chemical composition of fruits, being such attributes influenced mostly by acids, sugars and phenolic compounds (Borges & Pio, 2003).

Citrus fruits are considered to be sources of vitamins and fiber, and the consumption of fruit in natura or their juices provides the consumption of secondary metabolites that are rich in antioxidants such as ascorbic acid, phenolic compounds, flavonoids and limonóides (Pereira & Cardoso, 2012; Couto & Canniatti-Brazaca, 2010; Duzzioni et al., 2010; Barbasso et al., 2005). Antioxidants are chemical compounds that can prevent or reduce the oxidative damage of lipids, proteins and nucleic acids caused by reactive oxygen species, which include free radicals. The antioxidant compounds contained in fruits are able to react with the free radicals and decrease its harmful effects on the human body (Pereira & Cardoso, 2012; Couto & Canniatti-Brazaca, 2010; Duzzioni et al., 2010; Barbasso et al., 2005). The vitamin C found in citrus fruits helps to reduce the incidence of degenerative diseases, such as cancer, cardiovascular diseases, inflammation, brain dysfunctions and delaying the aging (Couto & Canniatti-Brazaca, 2010; Pimentel et al., 2005).

The Valencia sweet orange, the common group, is the most produced in Rio Grande do Sul and their harvest begins in late August early September and extends up to the month of December (Petry et al., 2012).

The Salustiana sweet orange can be harvested from mid-June to September (Oliveira & Scivittaro, 2011).

The ripe fruit can be kept in plants for a few months, without losing the commercial quality, feature a large amount of juice (> 50% of the fruit) and do not have seeds (Oliveira & Scivittaro, 2011).

The navel orange Lane late is a cultivar of late maturation, harvested between June and September. Its fruits can be kept in the trees, in excellent trading conditions, for a period of more the other cultivars of Umbilicus (Oliveira & Scivittaro, 2011).

Thus, this study aims to compare their organoleptic qualities and nutracêutica of fruit of three cultivars of oranges, as well as their waste with the aim of spreading the citrus fruit quality in the Middle High Uruguay of Rio Grande do Sul.

## MATERIALS AND METHODS

The experiment was conducted in the experimental area of the Polo of technological modernization of the Medium High Uruguay (PMTec), under geographic coordinates 27°22"S; 53°25"W, 480 m above sea level, in line with Faguense, municipality of Frederico Westphalen - RS. The climate of the region, according to the classification of Köppen is type Cfa, shrubland, without a dry season and temperature of the hottest month greater than 22°C. The collection of citrus PMTec was deployed in 2006 and is formed with a line of eight plants for each genotype grafted on the rootstock Trifoliata [*Poncirus trifoliata* (L.) Raf.].

The analysis of the quality of fruits were performed in the laboratory of analysis of foods of the Polo of technological modernization of the Medium High Uruguay (PMTec). The fruits were collected in the average height of 1.2 m, being withdrawn 20 fruits of each plant in the area of the plot in the four quadrants of the plant. The results were mixed, and the composite sample, we selected 10 fruits that have been washed and weighed. We evaluated the fruits of three varieties of orange [*Citrus sinensis* (L.) Osbeck], they were the cultivars Valencia, Lane Late, and Salustiana.

Subsequently, the fruits were cut in half, with the juice, and then separating the bark and pulp, in the fruits that it was also possible to split up the albedo. The results represent the mean of these ten fruits. Later dried up husks, pulps and albedo in an oven at 105 °C; milled into mill of knives; weighed out 5,0g of each sample, diluted in distilled water; filtered in gauze to 100 ml volumetric flask by completing the volume.

The maturity index was determined by the ratio between total soluble solids and titulabre acidity (TSS/TTA). The acidity titulabre was determined by volumetry of neutralization, as (IAL, 2008). The soluble solids were determined with the refractometer digital Tecnal, where dripped-if two drops of juice getting the results in Brix (IAL, 2008).

The determination of ascorbic acid was performed only in samples of juice, by the method of the Instituto Adolfo Lutz, with adaptations, which employs the potassium iodate to promote the oxidation of ascorbic acid (IAL, 2008). The determination of the content of carotenoids was performed in samples of bark, pulps and albedo, by the method of IAL (2008), with adaptations.

The determination of polyphenols was performed in samples of bark, pulp and albedo, by the method of Folin Ciocalteau, with adaptations, which is based on the reduction of the acids fosfomolibídico and fosfotunguístico in the presence of phenolic forms, with formation of complexes of blue coloration that absorb strongly between 620 and 800 nm (Katalinic et al., 2006; Minussi et al., 2003). the antioxidant activity was evaluated in samples of bark, pulp and albedo, using the method of kidnapping of free radicals of 2.2 diphenyl-1-picrilhidrazil (DPPH), with adaptations, which is based on a test where photometric the free radical DPPH, which features color intense purple solution in alcohol, is reduced by the presence of antioxidant molecules, forming the 2.2 diphenyl-1-picrilhidrazil, which It is colorless (Almeida et al., 2006).

Waste from the physical, chemical and qualitative characteristics of the fruits performed in the laboratory of analysis of foods are forwarded to the effluent treatment station of the Polo of technological modernization of the URI, where will be treated by the chemist: Pereira da Silva, CRQ no. 05100147, under his responsibility.

The results recorded for the different parameters of quality of fruits were subjected to analysis of variance (ANOVA). The results were also submitted to the Tukey test to identify differences between the averages and multivariate analysis to the study of correlation. It will be established the significance level of 5% probability.

#### **RESULTS AND DISCUSSION**

According to Volpe et al. (2002), in Brazil the harvest of citrus begins when the fruits reach a maturity index of at least 12.0. All cultivars presented within the content of commercial maturity used in Brazil (Table 1). The values are lower than those found by Couto & Canniatti-Brazaca (2010) for the Valencia orange (13.33) and higher than those found to Baía orange (6.67). Petry et al. (2012) have broken down the physics and chemistry of Valencia oranges, produced under organic and conventional cropping and harvested at different dates and observed values of ratio between 9.27 and 12.23. Being that the value shown in Table 1 shows itself within this range.

Yet, in Table 1, it can be noticed that the cultivar that showed higher levels of ascorbic acid was the Salustiana (40.71 mg.100 mL<sup>-1</sup>). Comparing it with the study by Pereira & Cardoso (2012) that examined the quality of tropical fruits and citrus fruit, where the citrus fruits of different species showed levels of ascorbic acid between 33.90 and 44.70 mg.100 mL<sup>-1</sup>, and the cultivars of orange Lane Late, Salustiana and Valencia showed levels within the range reported by Pereira & Cardoso (2012). Couto & Canniatti-Brazaca (2010) found for different cultivars of oranges and tangerines, between 21.47 mg.100 mL<sup>-1</sup> and 84.03 mg.100 mL<sup>-1</sup> of ascorbic acid, and in this case to the orange Valencia the authors found a ascorbic acid of 79.47 mg.100 mL<sup>-1</sup> and orange Baia of 80.03 mg.100 mL<sup>-1</sup>, whether hauling much of the results obtained for the cultivars analyzed in their work.

However, none of these studies report the degree of maturation of the fruit, since fruits less mature feature in higher content of ascorbic acid compared to mature fruit, which can be attributed to the action of enzymes such as ascorbate oxidase and cytochrome oxidase and fenolase (Yamada et al., 2012). One should take into account the place of origin of the samples, as shown Yamada et al. (2012), which examined the composition of the same species of citrus, with samples from different locations and it was observed that the ascorbic acid content ranged from 21.47 to 54.12 mg.100 mL<sup>-1</sup>, which represents a significant difference, which can be justified by the different conditions to which the fruits and plants were exposed once has its origins.

In Table 2 it can be seen that the higher content of carotenoids obtained in their study was  $0.64 \text{ mg}.100 \text{ g}^{-1}$  in albedo of cultivar Salustiana, and the lowest value was  $0.25 \text{ mg}.100 \text{ g}^{-1}$  in the bark of the cultivar Valencia. Among the studied cultivars presented the highest contents both in shell ( $0.63 \text{ mg}.100 \text{ g}^{-1}$ ) and the pulp ( $0.62 \text{ mg}100 \text{ g}^{-1}$ ) was to cultivate Lane Late. If compared their work with the study by Pereira & Cardoso (2012), where they were analyzed the pulps of various species of fruit, including citrus, realizes that the results are within the range obtained by this author,

0.05 to 0.70 mg.100 g<sup>-1</sup> for different species of citrus. The quantities of carotenoids found (Table 2) and of other studies performed with citrus fruits consider the fact that the carotenoids degrade com ease (Lourenço et al., 2013; Couto & Canniatti-Brazaca, 2010). Its contents are very low compared to levels found in other fruits and fall away much of the levels recommended dietary intake (Couto & Canniatti-Brazaca, 2010).

According to Table 3, it is observed that the peel of all cultivars showed levels of polyphenols significantly in excess of the pulp, except for the cultivar Salustiana, which showed no significant difference between the content of polyphenols present in the bark, 45.93 mg.100 g<sup>-1</sup>, and in the pulp 45.76 mg.100 g<sup>-1</sup>, presenting a considerable difference in albedo 53.01 mg.100 g<sup>-1</sup>. Among the cultivars, the Valencia showed the highest content of polyphenols in the pulp. In both cultivars that were unable to separate the albedo of the pulp, Lane Late and Salustiana, it was realized that the content of polyphenols is greater in albedo than in the pulp. Compared with the study by Pereira & Cardoso (2012), where they were analyzed the pulps of various species of fruits, including oranges, realizes that the results of total polyphenols obtained on their work, for the pulps, are within the range reported by this author for different citrus species studied, 30.00 to 76.10 mg.100 g<sup>-1</sup>.

In Table 4, it is possible that all cultivars had higher antioxidant activity in pulp than in the bark, being that the cultivar Salustiana presented the highest levels, 62.33% in the bark and 67.79% in the pulp, in addition to 64.19% in albedo. The cultivar of the orange Lane Late presented a percentage a little less, 52.52% in the bark and 57.75% in the pulp, Couto & Canniatti-Brazaca (2010) evaluated the antioxidant activity of some cultivars of citrus, finding values between 12.78% and 66.24%, while the lower grades corresponding to cultivars of tangerines and the highest values corresponding to cultivars of orange. If compared these results with those obtained on their job realizes that the values found to orange are in the same range of values. But you should take into consideration that the study of Couto & Canniatti-Brazaca (2010) evaluated the antioxidant activity in juices, not having examined the bark and pulp, therefore, the difference in values may be associated with different contents of phenolic compounds, among others (Couto & Canniatti-Brazaca, 2010).

Bernardes (2011) also evaluated the antioxidant activity of some species of fruit, finding an antioxidant activity of 51.88% for hulls and 59.87% for the pulp of orange. If compared these results with those found in their work realizes that Bernardes (2011) also found greater antioxidant activity in the pulp that the bark, being that

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Sample	TSS (°Brix)	TTA (%)	TSS/TTA (ratio)	Ascorbic acid content (mg.100 mL <sup>-1</sup> of juice)
Lanelate	$10.00\pm0.13b$	$0.68 \pm 0.12c$	14.70a	$33.92 \pm 0.07b$
Salustiana	10.66± 0.09a	$0.77 \pm 0.20a$	13.84a	$40.71 \pm 0.04a$
Valencia	$10.12\pm0.13b$	$0.72 \pm 0.22b$	14.05a	$38.51 \pm 0.05a$

 Table 1. Total soluble solids (TSS), total titulabre acidity (TTA), maturity index or ratio (TSS/TTA) and ascorbic acid content of three orange cultivars collected from the PMTec collection in Frederico Westphalen\*

\*Columns with different letters are significantly different at the Tukey test ( $p \le 0.05$ ).

Table 2. Total carotenoid content in three orange cultivars collected in the PMTec collection in Frederico Westphalen\*

Total carotenoid content (mg.100 g <sup>-1</sup> )					
Sample	Bark	Albedo	Pulp		
Lanelate	$0.63 \pm 0.02 \text{ aA}$	$0.31 \pm 0.02 \text{ bB}$	$0.62 \pm 0.01 \text{ aA}$		
Salustiana	$0.50 \pm 0.03 \text{ bB}$	$0.64 \pm 0.04 \text{ aA}$	$0.58 \pm 0.05 \text{ aA}$		
Valencia	$0.25 \pm 0.04 \text{ cB}$	**	$0.33 \pm 0.02 \text{ bA}$		

\*Columns with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). Lines with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). \*\* Not detected.

Table 3. Polyphenols in three orange cultivars collected in the PMTec collection in Frederico Westphalen\*

Polyphenols content (mg GAE.100 g <sup>-1</sup> )					
Sample	Bark	Albedo	Pulp		
Lanelate	$61.60 \pm 0.05 \text{ bA}$	$33.18 \pm 0.04 \text{ bB}$	$32.68 \pm 0.04 \text{ cB}$		
Salustiana	$45.93 \pm 0.06 \text{ cB}$	$53.01 \pm 0.03 \text{ aA}$	$45.76 \pm 0.17 \text{ bB}$		
Valencia	$84.18 \pm 0.13$ aA	**	$56.60 \pm 0.09 \text{ aB}$		

\*Columns with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). Lines with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). \*\*Not detected.

Table 4. Antioxidant activity in three orange cultivars collected in the PMTec collection in Frederico Westphalen\*

Antioxidant activity (% S.R.L.)						
Sample	Bark	Albedo	Pulp			
Lanelate	$52.52 \pm 0.13 \text{ bAB}$	$49.67 \pm 0.04 \text{ bB}$	57.75 ± 0.15 bA			
Salustiana	$62.33 \pm 0.03$ aA	$64.19 \pm 0.06 \text{ aA}$	$67.79 \pm 0.13$ aA			
Valencia	$59.39 \pm 0.11 \text{ abA}$	**	$63.21 \pm 0.20$ aA			

\*Columns with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). Lines with different letters are significantly different at the Tukey test ( $p \le 0.05$ ). \*\*Not detected.

their results were closer to those obtained for the cultivar Lane Late, 52.52% for hulls and 57.75% for the pulp (Bernardes, 2011).

Through the results obtained it is seen that the Valencia, Salustiana and Lane Late oranges possess considerable antioxidant activity and concentration of polyphenols in the composition of its fruits. Despite the results vary according to the environmental characteristics of cultivation, maturity and variety of fruits (Yamada et al., 2012; Couto & Canniatti-Brazaca, 2010; Duzzioni et al., 2010). Citrus are fruits that have high levels of total polyphenols, especially in its bark, which can lead to reuse their waste as a source of polyphenols fruits (Yamada et al., 2012; Couto & Canniatti-Brazaca, 2010; Duzzioni et al., 2010).

Usually the citrus fruits produced in Southern Brazil have higher acidity and lower *ratio* compared with fruits produced in the Southeast and Northeast regions of Brazil (Borges & Pio, 2003). Therefore, warmer climates provide more prompt production, higher content of juice, peel color less intense, higher total soluble solids content and a lower total titulabre acidity and consequently a higher *ratio* (Borges & Pio, 2003). It is expected that a subtropical climate, according to Borges & Pio (2003), which the citrus fruits have lower total soluble solids content and increased total titulabre acidity, and therefore lower *ratios*. The results found in this study agree in part with this trend.

According to the results obtained it is seen that the cultivars studied oranges have a considerable amount of ascorbic acid in your juice. They also have a considerable amount of polyphenols in their bark and pulp, and high antioxidant activity, being that the pulps showed higher contents. Therefore, the cultivars studied oranges collected in stage of commercial maturity, under these conditions, have chemical characteristics that enable the reuse of waste materials mainly as a source of polyphenols and as antioxidants.

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