

# Quality of Clementine Jam Influenced by Purée Pretreatment, Sugar Type and Pectin Addition

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## Summary

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The aim of this research was to investigate the influence of several parameters on quality of Clementine (*Citrus clementine*) jam. Thermal treated and nontreated Clementine purée, two types of sugar (sucrose and fructose) and three different amounts of added pectin were used for the jam producing. Accordingly, twelve jams were prepared and sorted in four groups, two of nontreated purées and two of treated purées, and each one with sucrose and fructose within.

The quality of jams was evaluated through gel strength measurement, sensory evaluation and level of polyphenols. The results of gel strength measurement showed that the treated purée jams were less firm than nontreated ones, the jams with sucrose were firmer than the ones with fructose and the gel strength of jams increased with amount of added pectin increase. Jams of nontreated purée contained higher level of polyphenols in comparison with jams of treated purée. Regarding to sensory evaluation, the nontreated purée jams were higher rated in comparison to treated ones. Furthermore, jams with fructose were better evaluated than jams with sucrose. Based on obtained results, it is revealing that nontreated purée jam with fructose and 0.7 % added pectin was the best sensory evaluated sample.

## Key words

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Clementine, jam, gel strength, sensory evaluation, phenolic compounds, pectin

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## Introduction

Citrus fruits, including Clementine (*Citrus clementine*), are very popular in human nutrition, mostly consumed as fresh fruit, but also as processed. Additionally, citrus fruits have received much attention, because of its interesting chemical composition, containing high amount of bioactive compounds, especially polyphenols, vitamin C, carotenoids and dietary fibers (Marlett, 1992; Marlett and Vollendorf, 1994; Craig, 1997; Del Caro et al., 2004; Tripoli et al., 2007; Wang et al., 2007; Wang et al., 2008). Many studies showed that those compounds have a beneficial effect on the human health (Wang et al., 2007) and great influence on reducing risk of some disease like cancer, cardiovascular and other diseases (Ejaz et al., 2006).

Citrus fruits (both the edible and inedible parts) are a particularly rich source of pectin, which has multiple biological activities, including glycemic and cholesterol level control (Baker, 1994; Seymour and Knox, 2002).

Citrus fruit consumption (Clementine so ever) in fresh condition is seasonally limited and as a fruit with many eligible characteristics, it can be processed into different products, including jams, juices, candied peels, etc. (Levaj et al., 2005).

Unfortunately, citrus peel is often waste material, obtained in juice industry (Rehman, 2006), but it can be added in citrus jams and additionally enrich sensory and nutritional characteristics of product while it is abundant source of pectin, natural flavonoids and contains higher amount of polyphenols compare to the edible portions (Rouseff et al., 1987; Braddock, 1995; Manthey and Grohmann, 2001; Chou and Huang, 2003; Wang et al., 2007; Wang et al., 2008). Jams are usually produced with high content of sugar, mostly sucrose, so producing low sugar jams with fructose could be nutritively more acceptable. Additionally, amount and type of sugar could influence on jam firmness so type and amount of added pectin as gelling agent has to be appropriate. Jam manufacture usually are based on frozen feedstock which could be thermally treated before freezing. Purpose of thermal pretreatment usually is inactivation of enzyme and reducing of microorganisms, but it could influence on many fruit components like pectic substances or polyphenols. Pectin, not only added but also pectic substances originate from fruit part of jam, is important factor in forming gel structure during jam production (Carbonell et al., 1991). Pectic substances present in all fruit as well as in Clementine are very complex group of polysaccharides and its ability to form gel is in dependence of its composition and structure (Seymour and Knox, 2002). Our interest in this research work was to investigate thermal impact of purée, besides sugar type and amount of added pectin, on the overall quality of low sugar Clementine jam. Quality was evaluated by jam's gel-strength measuring, sensory evaluation and additionally by determination of polyphenols level.

## Material and methods

### Samples

Fresh Clementine (*Citrus clementine*) was purchased in local market in Zagreb (Croatia) and used for the jam production. Food grade commercial sucrose and fructose were used, as well as low ester and amidated pectin.

Pulp and peel of Clementine were used for jam preparation. Fruits were hand-peeled. Edible parts were homogenized by blender (Zepter mixSy). One part of purée was heated till boiling, chilled and strained. Peel was chopped into pieces of 0.5 x 0.5 cm and was cooked three times in boiling water for ten minutes and cooled. After this preparation, purée and peel were frozen, and kept at  $-18^{\circ}\text{C}$ . They were defrosted at room temperature before the preparation of jams.

Jam was made according to the following recipe: 1000 g of product contained 460 g of Clementine purée and 390 g of sugar, and total dry matter was 45 %. All jams were defined with: one of two types of purée (thermal treated and no treated purée), one of two types of sugar (sucrose and fructose) and one of three different amounts (0.5 %, 0.7 % and 0.9 %) of pectin (jams produced with treated purée: further treated purée jams and with no treated purée: further non-treated purée jams).

Purée was cooked under atmospheric pressure to obtain approximately 20 % soluble solid matter and then sugar was added. Process of cooking was continued with stirring and pectin solution was added few minutes before soluble dry matter content was 45° Brix. Pieces of cooked peel were added in the end.

All twelve jams were put into glass jars and vials of Sulc pectinometer and stored under refrigeration conditions ( $7^{\circ} \pm 2^{\circ}\text{C}$ ) till analysis.

### Methods

Physicochemical parameters, dry matter, pH value, total acidity, reduced sugars and sucrose were determined in accordance with methods described by Tanner and Bruner (1979). Soluble dry matter was determined by measuring °Brix (Leica 7531 L refractometer).

Total phenols, flavonoids and nonflavonoids were determined using Folin-Cioacalciu colorimetric method (absorbance measuring at 750 nm) and the results were expressed as mg of gallic acid equivalents (GAE)/kg fresh weight. The formaldehyde precipitation was used to determine flavonoids and its amount was calculated as difference between total phenols and nonflavonoids (Ough and Amerine, 1988).

Pectic composition was determined by spectrophotometric method (IFU, 1964). Water-soluble, oxalate-soluble and alkali-soluble pectic fractions were extracted, quantitatively measured and calculated by calibration curve and expressed as mg of galacturonic acid equivalents (GA)/kg fresh weight.

All spectrophotometric measurements were performed by UV-VIS spectrophotometer UV-Vis Unicam β.

**Table 1.** Sensory evaluation sheet

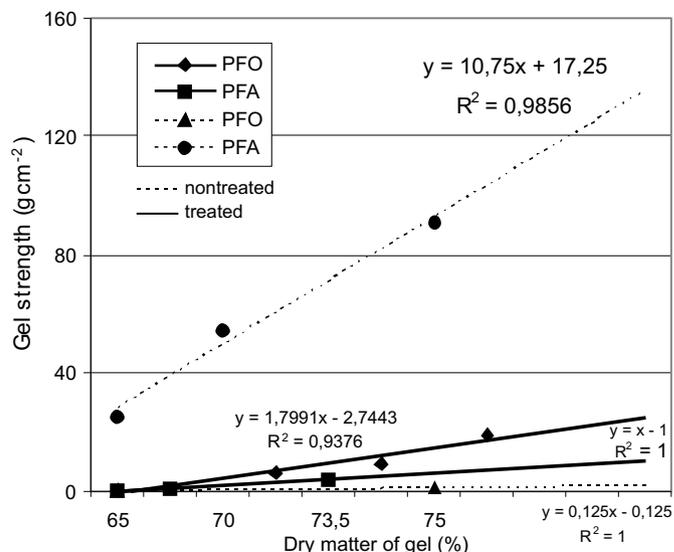
Sensory attributes	Sensory characteristic	Points
Taste	specific for fruit and product, intensive and harmonious	6
	specific for fruit and product, with less intensity	4-5
	still specific for product but less intensive, total impression is not markedly harmonious	2-3
Odour	taste of fruit with some different off-taste	1
	rotten taste, mould taste	0
	remarkably, specific and harmonious for fruit and product	4
	odour specific for fruit and product, but less intensive	3
	reduced odour, but still specific for product	2
Colour	no odour or slightly strange odour expressed	1
	rotten odour, mould odour	0
	specific and intensive colour for fruit and product	4
	specific with different intensity (too strong or too weak)	2-3
	not specific, too pale or too dark	1
Firmness (texture)	colour doesn't relate to relevant fruit	0
	optimal, easy to spread	6
	easy to spread, slightly strong or slightly weak with appearance of liquid	4-5
	not easy to spread, too strong or too weak	1-3
	totally inappropriate firmness	0

Pectic fractions were also used to investigate their ability to form gel (Levaj et al., 2003). Extracted pectic fractions were obtained from 20 g Clementine pulp according to mentioned method (IFU, 1964) and were used to make gels by adding 20 g sucrose and citric acid to adjust pH-value to 3.2. Gels were cooked under atmospheric pressure until certain dry matter was obtained. During the cooking, gel samples were taken for gel strength and dry matter measuring by refractometer and were kept in refrigerator for 24 hours. Gel strength ( $\text{g cm}^{-2}$ ) of gels and jams was measured by Sulc pectinometer (Sulc, 1983). Gels and jams were kept at room temperature at least for one hour before measuring.

In all jams sensory attributes of colour, odour, taste and firmness (texture) were evaluated by panel group which was consisted of eight members and with 20 - point system. Maximal points for taste and firmness (texture) were 6 and for colour and odour 4 (Table 1).

## Results and discussion

Thermal pretreatment of Clementine purée had slightly impact on physicochemical parameters. According to results shown in Table 2 thermal treated purée had a little bit higher dry matter, total sugars and pH value but a little bit lower total acidity than nontreated purée. The level of reducing sugars in treated purée was higher as a result of sucrose hydrolysis so the level of sucrose was very low. Soluble dry matter was almost the same as total dry matter. Additionally dry matter and sugar level in both purées was mostly in the same range as data in literature (Senser and Scherz, 1991).



**Figure 1.** Gel strength of gels prepared from oxalate-soluble (PFO) and alkali-soluble (PFA) pectic fractions from thermally nontreated and treated Clementine purée

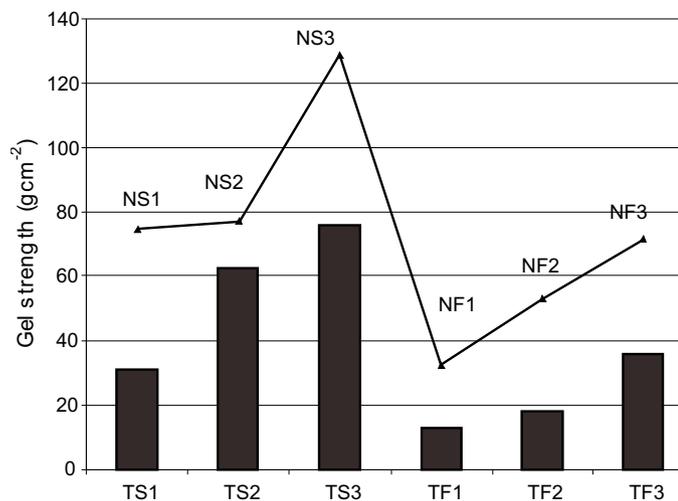
Content of pectic fractions showed remarkable differences between purées. In comparison to treated purée, nontreated purée showed higher content of oxalate-soluble pectic fraction (PFO) and alkali-soluble pectic fraction (PFA). Degradation of pectin molecule caused higher amount of water-soluble pectic fraction (PFW) in treated purée.

The ability of individual pectic fraction to form gel was investigated because it is not only dependant on level of present galacturonic acid in extract but also on composition and structure of extracted pectic fractions. Extracted pectic fractions were used to prepare gel. PFW didn't form gel in both purées (Figure 1) in spite of very high level of galacturonic acid in nontreated and even higher in treated purée (Table 2). Its composition and structure is usually responsible for its disability to form gel (Seymour and Knox, 2002; Levaj et al., 2003)

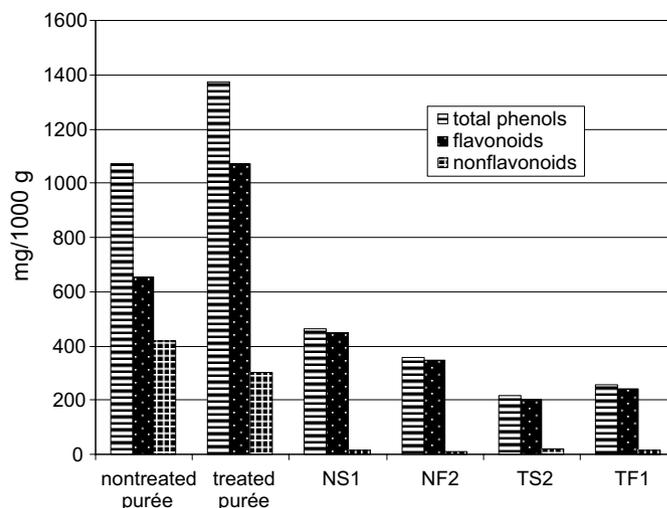
PFO and PFA from both purées formed gels and gel strength was increasing by increasing of dry matter although they behaved differently depending on purée type.

**Table 2.** Physicochemical parameters in Clementine purée

Parameters		Nontreated purée	Treated purée
Dry matter (%)	Soluble	12.75±0.14	13.25±0.09
	Total	12.80±0.32	13.32±1.29
pH		3.14±0.00	3.17±0.00
Total acidity (%)		1.53±0.03	1.25±0.03
Sugars (%)	Total	7.27±0.05	8.96±0.08
	Reducing	3.64±0.09	8.68±0.07
	Sucrose	3.45±0.04	0.27±0.00
Pectic fractions soluble in (mg GA/kg)	Water	2526.21±9.71	4346.46±101.78
	Oxalate	3006.80±228.15	1296.44±128.00
	Alkali	4909.71±33.98	735.79±71.11



**Figure 2.** Gel strength of Clementine jams (N: nontreated puree jams; T: treated puree jams; S: jams with sucrose addition; F: jams with fructose addition; 1: 0.5 % added pectin; 2: 0.7 % added pectin; 3: 0.9 % added pectin)



**Figure 3.** Polyphenols in purées and jams (N: nontreated puree jams; T: treated puree jams; S: jams with sucrose addition; F: jams with fructose addition; 1: 0.5 % added pectin; 2: 0.7 % added pectin)

**Table 3.** Sensory evaluation of Clementine jams

Sample		N <sup>a</sup> S <sup>b</sup> 1 <sup>c</sup>	NS2 <sup>c</sup>	NS3 <sup>c</sup>	NF <sup>b</sup> 1	NF2	NF3	T <sup>a</sup> S1	TS2	TS3	TF1	TF2	TF3
Average point for	Colour	31	29	28	30	29	30	25	27	28	31	30	30
sensory attribute	Odour	28	29	24	26	28	26	27	25	26	30	29	27
	Taste	35	33	31	29	40	39	36	38	34	39	36	37
	Firmness	40	39	35	37	41	39	36	37	36	36	37	39
Total score		16.75	16.25	14.75	16.5	17.25	16.75	15.5	15.9	15.5	17.0	16.5	16.6

<sup>a</sup> - N: nontreated puree jams; T: treated puree jams; <sup>b</sup> - S: jams with sucrose addition; F: jams with fructose addition; <sup>c</sup> - 1: 0.5 % added pectin; 2: 0.7 % added pectin; 3: 0.9 % added pectin

Gel with the highest firmness was made with PFA from nontreated purée in which fraction was found the highest level of galacturonic acid, too (Table 2). PFA from treated purée made very weak gel.

PFO fraction from nontreated purée made very weak gel even at high dry matter. PFO from treated purée made little bit firmer gel than PFA from treated purée and even PFO in nontreated purée (besides lower level of galacturonic acid).

Generally, it could be concluded that in nontreated purée PFA is more responsible while in treated purée PFO and PFA have similar responsibility for forming gel.

Furthermore, gel strength of jams from treated purée (Figure 2) was less firm than ones from nontreated purée in spite the same amount of added pectin. It means, that pectic substances present in Clementine purée really contribute to gel strength of jams. Evidently, PFA in nontreated purée had noticeable effect on gel strength of related jams. Additionally, jams with sucrose were two fold firmer than ones with fructose. Nevertheless, gel strength of jams linearly increased with amount of added pectin in all investigated cases (treated or nontreated purée, added sucrose or fructose).

All samples of Clementine jams were sensory evaluated by point system. According to obtained results (Table 3),

purée pretreatment and sugar type had an influence on sensory attributes of jams. Nontreated purée jams generally had higher total scores than treated purée jams, though treated purée jams got higher taste scores, due to thermal treatment impact on aroma compounds. Fructose jams mostly had better evaluated sensory attributes than ones with sucrose, especially for taste. In general, firmness of nontreated purée jams was better evaluated than firmness of treated purée jams. Mainly, the influence of the amount of added pectin on jam's firmness evaluation was dependant on type of purée and type of sugar. In nontreated purée jams with sucrose increase of added pectin influenced negatively firmness evaluation while in ones with fructose as well as in treated purée jams with fructose positively. In nontreated purée jams with sucrose this influence was not noticeable. Different evaluation of jam firmness related to added sugar was in accordance with previous statement about sugar influence on jam's firmness (Figure 2) because jam with very high firmness is not sensory appreciable.

Because all prepared jams could be divided into four groups (nontreated or treated purée, sucrose or fructose), the best sensory evaluated jams in each group were picked for additional quality control namely for polyphenols level measurement.

The results of physicochemical analysis in picked jams (results are not shown) were in accordance with recipe (dry matter, pH, total acidity and sugars).

Concerning the polyphenols (Figure 3), higher amount of polyphenols was determined in treated purée than in nontreated puree. It could happen because in some cases polyphenols in fresh fruit tissue is in such form that extraction conditions could not be sufficiently effective. Whereas during the thermal treatment fruit tissue passes through some structural changes that enhance effectiveness of extraction method.

Additionally, according to our results processing purée into jams caused considerable decrease of polyphenolic compounds. That means that further exposition of fruit tissue to high temperature during jams production had negative influence on polyphenols level. Moreover, higher level of polyphenols was determined in nontreated purée jams than in treated purée jams. It is important to take in account that decrease of polyphenols was also result of fruit level in jams (460 g purée/1000 g jams) not only of cooking process. In jams total phenols and flavonoid content was nearly the same, what is caused by almost complete degradation of nonflavonoids.

## Conclusion

The quality of Clementine jams was estimated by jam's gel-strength measuring, sensory evaluation and by the content of polyphenols.

Generally, all jams produced with treated purée were less firm than nontreated ones. Further, jams with fructose were less firm than the ones with sucrose. Based on sensory evaluation, the best evaluated was nontreated purée jam with fructose and 0.7 % of added pectin.

All jams had lower amount of polyphenols in comparison to purées and all treated jams had lower polyphenols than nontreated ones.

According to our results, thermal pretreatment didn't show particular positive influence on Clementine jam quality.

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