THE EFFECT OF RIPENING AND STORAGE ON PEACH PECTIN AND GEL STRENGTH OF RELATED JAMS

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Peaches were harvested in three stages of ripeness (threshold-mature – I, firm-ripe – II and ripe – III). Firm-ripe (II) peaches were stored for 9 days at 4 and 22 °C. The amount of water-soluble, oxalate-soluble and alkali-soluble pectin were extracted and quantitatively determined as galacturonic acid, neutral sugars were determined as glucose. Jams were made with all peach samples as well as gels from extracted fractions. Gel strength of jams and gels was measured.

Dry matter, sugar composition (amount of sucrose and reducing sugars) and pH value were determined and very little change was found during the ripening period and storage, too.

Alkali-soluble fraction was more abounding than water-soluble fraction. The oxalate-soluble pectic fraction was found to be a minor fraction in all peach samples. The amount of total pectin and the amount of alkali-soluble pectin fraction dropped markedly after the 1st stage of ripening, after the 2nd day of storage at 22 $^{\circ}$ C and slightly after the 6th day of storage at 4 $^{\circ}$ C.

The level of alkali-soluble pectin in total pectin was inversely proportional to the level of watersoluble pectic fraction, and the level of oxalate-soluble pectic fraction in total pectin content was fairly constant during the ripening and storage. Gel strength of jams and gels correlated very well with changes of the amount of alkali-soluble pectic fraction.

The extracted alkali-soluble pectic fraction was capable of forming an acidic gel. Extracted oxalate-soluble pectic fraction forms a gel in the presence of calcium and solid matter more than 77%.

It seems that the amount of alkali-soluble pectic fraction in peaches is responsible in producing peach jams according to gel strength.

Keywords: peach, pectic substances, jam, gel strength

The stage of maturity of a fruit at harvest influences the quality, development of aroma components and shelf-life (LIM & ROMANI, 1964; SALUNKHE et al., 1968; SPENCER et al., 1978; MEREDITH et al., 1989; LYON et al., 1993). Many studies on peach maturity at the time of harvest and on subsequent storage and ripening have focused on chemical composition (soluble solids, sugars and acids) and physical characteristics (weight, size and firmness) (LYON et al., 1993). Also, some of the studies were focused on pectin content and pectolitic enzymes activities during maturation and storage (BUESCHER & FURMANSKI, 1978; GLOVER & BRADY, 1995; ARTES et al., 1996; TIJSKENS & RODIS, 1997; HEDGE & MANESS, 1998; FERNANDEZ-TRUJILLO et al., 1998). Firmness is related to pectin composition of peach (FISHMAN et al., 1993). Harvesting and handling practices are important factors affecting eating quality of peaches (LYON et al., 1993),

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which is also due to pectin composition (BUESCHER & FURMANSKI, 1978). Composition of peach pectin depends on varieties and the stage of maturity of the harvested peaches (SHEWFELT, 1965; CHAPMAN & HORVAT, 1990; FISHMAN et al., 1993) and it also influences textural characteristics of some peach products (SHEWFELT, 1965). LEVI and co-workers (1988) confirmed the contribution of the pectins to the structural-textural characteristics of fruit products in general and dehydrated fruits in particular. CLAYPOOL (1974) determined correlations between reduced firmness of canned fruit and higher fruit acidity, which is often measured in less mature fruit. Jam is one of the usual peach products and it is essentially a pectin gel in which the final texture depends on the adequate sugar-acid-pectin balance, but also on the structure and composition of fruit tissues (CARBONELL et al., 1991). Pectin substances as constituents of fruit cell walls affect the firmness of peach jams, especially if jams are prepared without adding commercial pectin (LEVAJ et al., 2000). CARBONELL and co-workers (1991) suggested that peach tissues have a greater capacity to «incorporate» sugar and reinforce pectin gel. Generally, textural characteristics of jams are important for their overall acceptability to the consumer (RAPHAELIDES et al., 1996).

Our objective was to elucidate the effect of pectin constitution of peaches (cv. Red Haven) at three stages of ripening (firmness) and during a 9-day storage of firm-ripe peaches at 4 and 22 $^{\circ}$ C on the gel strength of related jams and gels.

1. Materials and methods

1.1. Samples and preparation

Peaches (cv. Red Haven) were hand-picked at three stages of ripening (thresholdmature – I, firm-ripe – II, ripe – III) at a commercial orchard near Zadar, Dalmatia, Croatia in 1999. Firm ripe (II) peaches were stored for 2, 4, 6 and 9 days at 4 and 22 $^{\circ}$ C.

Peaches were peeled, than mesocarp was cut into small pieces and immersed immediately into liquid nitrogen and stored at -18 °C in sealed polyethylene bags until the analysis.

1.2. Methods

Dry matter was determined by drying at 105 °C to the constant mass and pH value by pH-meter.

Reducing sugars and sucrose were determined by the Luff-method (TANNER & BRUNNER, 1979).

1.2.1. Pectin extraction and carbohydrate analysis. Water-soluble, oxalate-soluble and alkali-soluble pectic fractions were extracted and quantitatively determined as galacturonic acid (GalA) (IFU, 1964). In pectin extracts, neutral sugars were determined as glucose (NS) by using phenol-sulphate method (DUBOIS et al., 1956) and the Scott method (SCOTT, 1979), as described previously (FISHMAN et al., 1993).

1.2.2. Gel and jam preparation. Water-soluble, oxalate-soluble pectic fractions and residue after extraction of water-soluble and oxalate-soluble fractions (this residue put under alkali extraction yield alkali-soluble fraction) obtained from 40 g of peach tissue, according to IFU method (1964), were used to make gels by adding 40 g of sucrose and citric acid, 10% (w/v), to adjust initial pH-value in peach samples. CaCl₂, 10% (w/v), was added to the oxalate soluble pectin fraction.

Jams and gels were prepared under laboratory conditions under atmospheric pressure. Peach pieces were homogenized in laboratory blender. Mass proportion of fruit (peach pulp) to the added sucrose before cooking (evaporating) was 1:1 (w/w). No commercial pectin was added.

Mixtures were cooked in beakers and continuously stirred while heating until the mixture reached 60, 65, 70 and 75% dry matter. When the desired dry matter content was reached, vials of Sulc pectinometer were filled with 5 ml of those gels or jams and kept at 4 °C for 24 h. Gel strength (g cm⁻²) of gels and jams was measured (SULC, 1983). Gels and jams were kept at room temperature for at least one hour before measuring.

1.2.3. Statistical analysis. All measurements were carried out in 3 independent replicates. ANOVA was used to determine significant differences.

2. Results

Dry matter, pH value and sugar composition (the amount of sucrose and reducing sugars) changed very little during ripening as well as during storage (Table 1). Dry matter changed between 10.03 and 13.64% and pH between 3.63 and 3.97. BROVELLI and co-workers (1998) reported similar results for Tropic-Beauty peaches. Reducing sugars content was generally about 2% and that of sucrose was generally about 6 to 7%. Similar results of Monroe peaches were reported earlier (CHAPMAN & HORVAT, 1990).

The amount of pectin (Table 2) depended on the stage of ripening and storage time and generally was higher than 0.6% (galacturonic acid + neutral sugars), except in too ripe peaches (stored for 4, 6 and 9 days at 22 °C), which is in the same proportions as reported previously (LEVI et al., 1988). Alkali-soluble pectic fraction was more abundant than that of water-soluble, and oxalate-soluble fraction was found to be a minor pectic fraction in all peach samples and was between 10 and 20% of the total pectin (see Fig. 1). LEVI and co-workers (1988) reported 16% for this fraction. Also, it changed very little during ripening and storage.

| Parameter | S | tages of ripenes: | s | | | | Days of | storage at | | | |
|-------------------------|------------------|-------------------|------------------|------------------|------------------|-----------------|-----------------|------------------|------------------|------------------|------------------|
| | | | | | 4 | c | | | 22 | °C | |
| | I | п | Ш | 2 | 4 | 9 | 6 | 2 | 4 | 9 | 6 |
| Dry matter (%) | 12.19 ± 0.27 | 11.78 ± 0.11 | 11.95 ± 0.05 | 13.16 ± 0.03 | 13.13 ± 0.01 | 11.99 ± 0 | 12.77 ± 0.35 | 10.03 ± 0.35 | 11.31 ± 0.08 | 10.28 ± 0.08 | 13.64 ± 0.08 |
| pH value | 3.63 ± 0.01 | 3.68 ± 0 | 3.97 ± 0.01 | 3.86 ± 0.05 | 3.67 ± 0.02 | 3.72 ± 0 | 3.74 ± 0 | 3.72 ± 0.01 | 3.76 ± 0.02 | 3.87 ± 0.02 | 3.81 ± 0 |
| Reducing | 1.66 ± 0.01 | 2.33 ± 0.04 | 1.98 ± 0.04 | 2.53 ± 0.02 | 2.03 ± 0.02 | 2.26 ± 0.17 | 2.17 ± 0.02 | 2.07 ± 0.04 | 2.22 ± 0.01 | 2.33 ± 0.01 | 2.82 ± 0.05 |
| sugars (%) | | | | | | | | | | | |
| Sucrose (%) | 6.78 ± 0.11 | 5.96 ± 0.01 | 4.49 ± 0.29 | 6.81 ± 0.29 | 7.80 ± 0.08 | 6.83 ± 0.21 | 6.92 ± 0.05 | 5.33 ± 0.16 | 6.42 ± 0.06 | 6.24 ± 0.13 | 6.81 ± 0.01 |
| Total sugars (%) | 8.79 ± 0.09 | 8.67 ± 0.04 | 6.70 ± 0.24 | 9.69 ± 0.08 | 10.30 ± 0.15 | 9.44 ± 0.09 | 9.44 ± 0.07 | 7.68 ± 0.10 | 8.97 ± 0.04 | 8.89 ± 0.10 | 9.98 ± 0.03 |
| ^a Each value | crepresents the | three three | e replicates | | | | | | | | |

Table 1. Results of dry matter, sugar and pH^a



Fig. 1. Pectin fractions expressed as percentage of total pectin content in peach samples obtained by IFU method. A: at three stages of maturity; B: stored at 4 °C; C: stored at 22 °C. □: Pectin fraction soluble in water; \overline{D}: pectin fraction soluble in oxalate; ■: pectin fraction soluble in alkali

| | | | Extractant | Sugar composition (mg/100 g) | | searces of themese | | | |
|-----------------|-------------------|-------------------|-------------------|---------------------------------|-------------------|--------------------|------------------|-------------------|------------------|
| | | | | | I | Π | п | | |
| | | | (PFW) | $GalA^b$ | 54.9 ± 4.79 | 55.2 ± 5.49 | 103.8 ± 9.72 | | |
| | | | water | NSc | 156.1 ± 15.45 | 213.0 ± 12.96 | 175.5 ± 8.62 | | |
| | | | (PFO) | GalA ^b | 41.4 ± 3.77 | 30.6 ± 1.80 | 46.5 ± 1.88 | | |
| | | | oxalate | | | | | | |
| | | | | NSc | 17.4 ± 1.56 | 37.5 ± 1.23 | 50.3 ± 3.23 | | |
| | | l | (PFA) | $GalA^b$ | 242.9 ± 2.12 | 169.9 ± 1.42 | 131.5 ± 2.16 | | |
| | | | alkali | | | | | | |
| | | I | | NS° | 263.4 ± 16.86 | 175.3 ± 4.47 | 126.6 ± 7.31 | | |
| Extractant | Shear | | | | Davs | of storage at | | | |
| | composition - | | | 4 °C | | D | 10 | 2 °C | |
| | (mg/100 g) | 2 | 4 | 9 | 6 | 2 | 4 | 9 | 6 |
| (PFW) | $GalA^b$ | 42.0 ± 1.11 | 47.7 ± 4.59 | 39.6 ± 1.29 | 47.0 ± 3.94 | 98.8± 3.05 | 37.1 ± 3.0 | 33.7 ± 2.42 | 44.1 ± 2.14 |
| water | | | | | | | | | |
| | NSc | 196.7 ± 7.03 | 224.6 ± 1.09 | 221.7 ± 7.72 | 253.1 ± 16.09 | 267.1 ± 18.7 | 1 132.0±12.30 | 121.9 ± 10.16 | 159.7 ± 11.5 |
| (PFO) | GalA ^b | 25.2 ± 1.80 | 28.2 ± 0.82 | 19.4 ± 0.63 | 20.0 ± 0.56 | 28.3 ± 1.25 | 25.1 ± 1.74 | 24.1 ± 1.77 | 25.3 ± 1.80 |
| oxalate | | | | | | | | | |
| | NSc | 24.2 ± 1.90 | 31.3 ± 4.79 | 28.5 ± 2.32 | 31.7 ± 0.22 | 24.2 ± 2.58 | 29.6 ± 2.25 | 30.6 ± 2.69 | 37.5 ± 2.80 |
| (PFA) alkali | GalA ^b | 137.2 ± 4.66 | 180.1 ± 3.84 | 225.6 ± 12.49 | 148.9 ± 11.17 | 278.6 ± 26.6 | 8 80.7 ± 7.24 | 55.1 ± 4.74 | 60.1 ± 0.49 |
| | NSc | 136.6 ± 12.86 | 143.5 ± 12.94 | 172.5 ± 3.99 | 139.3 ± 13.49 | 149.0 ± 7.62 | 122.4 ± 3.10 | 125.3 ± 11.84 | 89.5 ± 5.46 |

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During ripening (Table 2) the amount of alkali-soluble pectic fraction expressed as GalA decreased, as reported earlier (FISHMAN et al., 1993), and water-soluble pectic fraction increased. During storage at 4 °C, the amount of alkali-soluble pectic fraction had an increasing trend until the 6th day and during storage at 22 °C until the 2nd day and then markedly dropped. FISHMAN and co-workers (1993) investigated Redskin peaches during storage at 22 °C and obtained a similar increase of alkali-soluble pectic fraction between the 1st and the 3rd days of storage. The constitution of cell walls and many biochemical processes that occur in peach cell wall might influence the increase of the amount of alkali-soluble pectic fraction in that period. Pectins are associated with hemicellulose in the peach cell walls (HEDGE, 1996), therefore, this phenomenon could be explained by a certain enzymatic activity during ripening. Polygalacturonases depolymerize de-esterified pectin only (FISHMAN, 2000). The investigation of GLOVER and BRADY (1995) showed that there is a phase between the mature and ripe stage (classified by penetrometer) when total pectinesterases content is high. DOWNS and coworkers (1992) showed that the increase in exopolygalacturonase activity occurs late in ripening. There is a relatively high protease activity during peach mesocarp development (GALLESCHI et al., 1991) and pectin is also associated with protein (KNEE, 1973). Results obtained on strawberry indicate continuous synthesis of polyuronides during ripening (HUBER, 1984). The theory that synthesis of pectin substances was continued during maturing, ripening and senescence was known earlier, too (TOWLE & CHRISTENSEN, 1973).

Results of the amount of neutral sugars in oxalate-soluble pectic fraction were almost 10 times lower than those in the alkali-soluble pectic fraction, which was to be expected because oxalate-soluble pectic fraction was found to be a minor fraction in all peach samples. However, it could be observed that the amount of neutral sugars in oxalate-soluble pectic fraction increased during the investigated period of ripening and during storage at 22 °C, as it was reported earlier (FISHMAN et al., 1993).

In alkali-soluble pectic fraction, neutral sugars during ripening and during storage at 22 °C decreased and during storage at 4 °C increased, but the level of neutral sugars in pectin increased with the duration of storage and ripening, which was also reported earlier (FISHMAN et al., 1993).

Level of neutral sugars is the highest in water-soluble pectic fraction, as expected.

Results in Fig. 1 show that percentage (level) of water-soluble pectic fraction in total pectin increased during ripening and storage, and it was inversely proportional to the percentage of alkali-soluble pectic fraction in all investigated samples. In all investigated samples, alkali-soluble pectic fraction also had the highest level in total pectin. Figure 1 shows that in threshold-mature peaches, and in peaches stored 2 days at 22 °C and 6 days at 4 °C the percentage of alkali-soluble pectic fraction in total pectin was the highest and then decreased, whereas the amount of oxalate-soluble pectic fraction (Table 2) and also its percentage in total pectin (Fig. 1) was fairly constant.



Fig. 2. Gel strength of jams and gels prepared from peaches at three stages of maturity (A) then from peaches stored at 4 °C (B) and 22 °C (C) compared to concentration of pectin fraction soluble in alkali (PFA) obtained by IFU method. ▲: Gel (65% dry matter); ■: jams (65% dry matter); ●: pectin fraction soluble in alkali



Fig. 3. Effect of jam's dry matter on the gel strength. A: peach stored at 4 °C; B: peach stored at 22 °C for nine days. ♦: 1st day; ■: 2nd day; □: 4th day; ▲: 6th day; ●: 9th day

| Table 3. Correlation between PFA and | d gel strength | of gels and jams | with 65% DM in diffe | erent samples |
|--------------------------------------|----------------|------------------|----------------------|---------------|
|--------------------------------------|----------------|------------------|----------------------|---------------|

| Samples | Correlatio | on coefficient (r) |
|---|--------------|--------------------|
| | gel | marmalade |
| Different stage of ripeness | 0.79 | 0.81 |
| Different duration of storage at 4 °C Different duration of storage at 22 °C | 0.56 0.89 | 0.96 0.98 |

| Storage temperature | Source of variation | SS | df | MS | f | F _{crit} |
|------------------------|---------------------|----------|----|---------|-------|-------------------|
| | Storage days | 671.83 | 3 | 223.94 | 2.93 | 3.86 |
| 4 °C | Dry matter of jams | 10214.50 | 3 | 3404.83 | 44.54 | 3.86 |
| | Error | 688.07 | 9 | 76.45 | | |
| | Total | 11574.40 | 15 | | | |
| | Storage days | 536.61 | 3 | 178.87 | 9.69 | 3.86 |
| 22 °C | Dry matter of jams | 3265.58 | 3 | 1088.53 | 58.95 | 3.86 |
| | Error | 166.17 | 9 | 18.46 | | |
| | Total | 3968.36 | 15 | | | |

Table 4. Variance analysis of data in Fig. 3

Water-soluble and oxalate-soluble pectic fractions and the solid residue after these extractions were used to make gels. Water-soluble pectic fraction was not able to form gel. Pectic fraction soluble in oxalate formed gel only in the presence of calcium ions and solid matter higher than 77.5% (data not shown). Gels were formed with the residue (after extraction of water-soluble and oxalate-soluble pectic fractions). Gel strength of jams and gels and the amount of alkali-soluble pectic fraction markedly decreased between the 1st and the 2nd stage of maturity (Fig. 2). The peaches stored for 6 day at 4 °C gave the highest gel strength of jam and gel. The amount of alkali-soluble pectic fraction was the highest, as well. Peaches stored at 22 °C had the highest results at the 2nd day of storage. Correlation coefficient between the amount of alkali-soluble pectic fraction and gel strength of jams and gels was high (Table 3).

Generally, gel strength of jams made of peaches stored at 4 °C did not change significantly with storage time and was firmer than those made of peaches stored at 22 °C (Fig. 3). After the 2nd day, storage at 22 °C had negative influence on gel strength of jams (Table 4).

3. Conclusions

During the investigated period of ripening and storage, the basic physical and chemical parameters changed very little.

Alkali-soluble pectic fraction was more abounding than that of water-soluble, and oxalate-soluble pectic fraction was found to be a minor fraction in all peach samples.

During the investigated period of ripening and storage at 22 °C, the amount of total pectin as well as the amount of alkali-soluble pectic fraction decreased with the stage of ripening and duration of storage after the 2nd day. Between the 1st (the day of harvesting the firm-ripe peaches) and 2nd day a significant increase was observed.

During storage at 4 °C, the highest amount of total pectin and alkali-soluble pectic fraction occurred in peaches stored for 6 days and then decreased. The level of water-soluble pectic fraction changed inversely with the level of alkali-soluble pectic fraction.

Gels could be formed only with residue after extraction of water-soluble and oxalate-soluble pectic fractions (alkali soluble fraction).

Gel strength of jams and gels decreased markedly between the 1st and the 2nd stages of ripening. Significant changes were observed during storage at 22 °C, while no significant changes were observed during storage at 4 °C.

There was a high correlation between gel strength and concentration of alkalisoluble pectic fraction. It seems that the amount of alkali-soluble pectin fraction in peaches is responsible in producing peach jams according to gel strength.

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