Manapal and Marion fruits harvested on May 24 were rated for placental breakdown 6, 9, 12, and 15 days after turning. No pressure had been applied to these fruits, since no firmness measurements were made. Marion showed more placental breakdown than Manapal (Table 2) at each period. Manapal showed a gradual increase over the 6 to 15 day period, while Marion reached a maximum at 12 days (interaction not significant). Since no pressure had been applied, these results indicate that the breakdown shown in Table 1 was not entirely due to the firmness measurements. A comparison of the values in the two tables indicates that the pressure applied during the firmness measurement increased the amount of breakdown.

It is not felt that the placental breakdown is due to bruising during handling, since the fruits were handled carefully throughout the harvesting, transporting, and daily sorting. All this handling was done during the mature-green stage which is the stage McCollouch found to be quite unsusceptible to internal bruising. After reaching the turning stage, the fruits were held in a single layer without further movement until analyzed.

A correlation analysis of firmness values and breakdown ratings of 30 Marion fruits at the November 3 harvest showed no significant correlation between firmness and breakdown. The same result was found using Indian River fruits.

**Summary**

Four varieties and a breeding line were evaluated for firmness and placental breakdown in the spring and fall of 1961. The Marion variety was found to be softer and Step 390 firmer than Indian River, Manapal and Homestead. Marion had more and Step 390 less placental breakdown than the other varieties.

The breakdown was found even when no pressure was applied (as in the firmness determinations) and increased during the period from 6 to 15 days after turning. No correlation was found between firmness and the breakdown.

**Literature Cited**

The purpose of this paper is to show the effect of maturity on the characteristics of pectins extracted from peel, membrane, and juice sacs such as jelly grade, methoxyl content, and viscosity. Also, those characteristics which are of importance to any potential commercial producers of pectin in Florida are evaluated.

**Experimental Procedure**

*Preparation of Samples.*—Alcohol-insoluble solids (AIS) were prepared from peel, membrane, and juice sacs of Valencia oranges as described by Rouse et al. (14). Oranges of the Valencia variety were picked each month (December through June) during both the 1959-60 and 1960-61 seasons from the same trees in Block II at the Citrus Experiment Station. These trees were in a healthy and vigorous condition.

The AIS of similar components for each growing season were combined according to months and extracted for 1 hr. at 90° C. using a ratio of AIS to distilled water, 1:80, and a ratio of AIS to Zeo-Karb, 8:10 (9). This is equivalent to an acid extraction with pH 2.4 to 2.6. The ratio of water to solids was maintained throughout the extraction period. The mixture was then cooled to 60° C. in running tap water. After centrifugation, the liquid was decanted into a prepared Buchner funnel with gentle suction. The prepared mat on the Buchner funnel consisted of Cenco No. 13255 filter paper coated with 20 g. of Standard Super Cel. Solids from the centrifugation were releached with a small quantity of 40° C. distilled water, recentrifuged, and filtered. The combined filtrates were precipitated in 2 volumes of 99% isopropyl alcohol, washed 2 times in 80% alcohol, and finally in 99% alcohol. The washed isopropyl alcohol precipitates were dried under vacuum at 60° C. for 12 hr.

*Methods of Analysis.*—Although yields of pectin are based upon the amount of alcohol precipitate, the purity of these precipitates was determined as percentage of anhydrogalacturonic acid (AGA) (13). Values for equivalent weights and methoxyl contents were determined by the procedures of Owens et al. (12). Data for methoxyl contents were calculated on an AGA basis.

Relative viscosity was measured on a 0.5% solution of alcohol precipitate at 26° C. with an Ostwald viscometer (6).

Alcohol precipitates were ashed in a muffle furnace at a temperature no greater than 525° C. to constant weight.

Jelly grade of the pectin was determined by measuring the jelly strength of standard 65% soluble solids jellies with the Delaware Jelly-Strength Tester (1, 2). Jellies were prepared by the excess-acid method and allowed to set 24 hr. before testing.

Jelly unit is the resulting figure when multiplying yield of pectin by the jelly grade (7).

**Results and Discussion**

The effects of Valencia orange maturity as reflected by the various physical and chemical values of the extracted pectins from peel, membrane, and juice sacs are presented in Tables 1, 2, and 3, respectively. Rouse et al. (14) have shown that the average distribution of the component parts in the Valencia whole orange during the period between December and June was approximately 20% peel, 10% membrane, 20% juice sacs, 49% juice, and 1% seeds.

*Yield as Alcohol Precipitate.*—The yield of pectin from the peel (23.2 to 27.8%) and membrane (31.4 to 34.8%) remained practically constant throughout the maturation period, whereas the yield from juice sacs increased from 19.9% (December) to 24.9% (February) and gradually decreased to 17.5% (June). Gaddum (3) reported yields of total extracted pectins from Valencia orange albedo (18.2 to 30.2%) and pulp (14.8 to 30.4%) for an 8 month growth period. Purity of pectin was usually expressed as pectic acid or calcium pectate, and Gaddum showed that the total extracted pectins from albedo and pulp ranged between 84.47 and 90.84% pectic acid.

Pectins extracted from juice sacs were lowest in purity ranging from 79 to 88% as AGA, while the pectins from peel and membrane ranged from 86 to 97% AGA. Purity of the alcohol precipitates followed no correlation with seasonal growth of the Valencia oranges.

*Degree of Methylation and Equivalent Weight.*—For years the value of the methoxyl content of pectin has been controversial. It has been used to indicate purity and jellying power of pectin. Actually by knowing the degree of esterification, the food processor is able to determine the setting time of the pectin, the combining power with metallic ions, and the ability of the pectin to form either low-solids or normal gels.

The average values of the percent methoxyl during the 7-month season for peel, membrane, and juice sacs were 10.07, 10.95, and 10.50 respectively, indicating highly methylated pectins. Methoxyl contents varied with the purity of the alcohol precipitates and are reported on an AGA.
Table 1. Relationship of Valencia orange maturation to pectin values of the peel.

<table>
<thead>
<tr>
<th>Sampling Months</th>
<th>December</th>
<th>January</th>
<th>February</th>
<th>March</th>
<th>April</th>
<th>May</th>
<th>June</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield—%</td>
<td>27.8</td>
<td>23.2</td>
<td>24.8</td>
<td>27.0</td>
<td>23.2</td>
<td>24.9</td>
<td>23.8</td>
<td>25.0</td>
</tr>
<tr>
<td>Jelly grade</td>
<td>218.5</td>
<td>202.5</td>
<td>212.1</td>
<td>201.5</td>
<td>189.8</td>
<td>201.6</td>
<td>213.1</td>
<td>205.6</td>
</tr>
<tr>
<td>Jelly units</td>
<td>60.7</td>
<td>47.0</td>
<td>52.6</td>
<td>54.4</td>
<td>54.0</td>
<td>50.2</td>
<td>50.7</td>
<td>51.4</td>
</tr>
<tr>
<td>Purity as AGA1—%</td>
<td>95.0</td>
<td>92.0</td>
<td>97.0</td>
<td>89.0</td>
<td>92.0</td>
<td>89.0</td>
<td>86.0</td>
<td>91.4</td>
</tr>
<tr>
<td>Relative viscosity</td>
<td>10.5</td>
<td>13.2</td>
<td>8.9</td>
<td>7.4</td>
<td>6.0</td>
<td>6.2</td>
<td>7.3</td>
<td>8.5</td>
</tr>
<tr>
<td>Equivalent weight</td>
<td>1018.3</td>
<td>1209.5</td>
<td>1075.3</td>
<td>880.3</td>
<td>1018.3</td>
<td>920.8</td>
<td>1020.4</td>
<td>1020.4</td>
</tr>
<tr>
<td>Ash—%</td>
<td>2.02</td>
<td>--</td>
<td>1.58</td>
<td>--</td>
<td>1.87</td>
<td>--</td>
<td>1.71</td>
<td>1.80</td>
</tr>
<tr>
<td>Methoxyl2—%</td>
<td>10.05</td>
<td>10.11</td>
<td>10.77</td>
<td>9.75</td>
<td>9.61</td>
<td>9.94</td>
<td>10.24</td>
<td>10.07</td>
</tr>
</tbody>
</table>

1  Anhydrogalacturonic acid in alcohol precipitate.
2  Calculated on AGA basis.

Table 2. Relationship of Valencia orange maturation to pectin values of the membrane.

<table>
<thead>
<tr>
<th>Sampling Months</th>
<th>December</th>
<th>January</th>
<th>February</th>
<th>March</th>
<th>April</th>
<th>May</th>
<th>June</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield—%</td>
<td>33.9</td>
<td>34.0</td>
<td>32.5</td>
<td>34.8</td>
<td>34.1</td>
<td>33.6</td>
<td>31.4</td>
<td>33.5</td>
</tr>
<tr>
<td>Jelly grade</td>
<td>363.1</td>
<td>313.3</td>
<td>333.3</td>
<td>299.5</td>
<td>285.7</td>
<td>292.1</td>
<td>310.2</td>
<td>313.9</td>
</tr>
<tr>
<td>Jelly units</td>
<td>123.1</td>
<td>106.5</td>
<td>106.3</td>
<td>104.2</td>
<td>97.4</td>
<td>98.1</td>
<td>97.4</td>
<td>105.0</td>
</tr>
<tr>
<td>Purity as AGA1—%</td>
<td>93.0</td>
<td>89.0</td>
<td>96.0</td>
<td>97.0</td>
<td>94.0</td>
<td>92.0</td>
<td>92.0</td>
<td>93.3</td>
</tr>
<tr>
<td>Relative viscosity</td>
<td>16.7</td>
<td>18.4</td>
<td>15.6</td>
<td>15.9</td>
<td>12.8</td>
<td>10.9</td>
<td>13.6</td>
<td>14.8</td>
</tr>
<tr>
<td>Equivalent weight</td>
<td>1290.0</td>
<td>1172.6</td>
<td>1066.0</td>
<td>1066.0</td>
<td>967.1</td>
<td>943.4</td>
<td>1075.2</td>
<td>1077.2</td>
</tr>
<tr>
<td>Ash—%</td>
<td>1.64</td>
<td>--</td>
<td>1.67</td>
<td>--</td>
<td>0.87</td>
<td>--</td>
<td>1.48</td>
<td>1.42</td>
</tr>
<tr>
<td>Methoxyl2—%</td>
<td>11.46</td>
<td>11.35</td>
<td>10.40</td>
<td>10.45</td>
<td>10.79</td>
<td>11.02</td>
<td>11.16</td>
<td>10.95</td>
</tr>
</tbody>
</table>

1  Anhydrogalacturonic acid in alcohol precipitate.
2  Calculated on AGA basis.

basis (Tables 1, 2, and 3). This is in agreement with Gaddum (3), who showed that the degree of methylation of acid-extracted pectins were of normal value throughout progressive maturity. Except for the pectin extracted from the January sample of juice sacs (Table 3), which contained 12.01% methoxyl, the pectins extracted from the membrane were slightly higher in methoxyl than the pectins from the other 2 components during the maturation period, ranging from 10.40 to 11.46%.

Unless corrections can be applied (12), equivalent weight results are usually not completely reliable. Olsen et al. (11) pointed out that the equivalent or combining weight decreased proportionately to progressive demethylation of the pectin and that setting time of pectin jellies and the effects of salts are a function of the pectin's
Table 3. Relationship of Valencia orange maturation to pectin values of the juice sacs.

<table>
<thead>
<tr>
<th>Sampling Months</th>
<th>December</th>
<th>January</th>
<th>February</th>
<th>March</th>
<th>April</th>
<th>May</th>
<th>June</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield—%</td>
<td>19.9</td>
<td>20.4</td>
<td>21.9</td>
<td>21.4</td>
<td>22.1</td>
<td>18.9</td>
<td>17.5</td>
<td>21.2</td>
</tr>
<tr>
<td>Jelly grade</td>
<td>236.4</td>
<td>210.3</td>
<td>198.2</td>
<td>204.7</td>
<td>209.7</td>
<td>236.4</td>
<td>228.5</td>
<td>222.0</td>
</tr>
<tr>
<td>Jelly units</td>
<td>47.0</td>
<td>49.0</td>
<td>49.4</td>
<td>49.9</td>
<td>56.3</td>
<td>41.7</td>
<td>40.0</td>
<td>46.6</td>
</tr>
<tr>
<td>Purity as AGA—%</td>
<td>80.0</td>
<td>83.0</td>
<td>79.0</td>
<td>85.0</td>
<td>88.0</td>
<td>82.0</td>
<td>82.0</td>
<td>82.7</td>
</tr>
<tr>
<td>Relative viscosity</td>
<td>11.2</td>
<td>9.5</td>
<td>6.9</td>
<td>6.8</td>
<td>7.4</td>
<td>9.8</td>
<td>8.4</td>
<td>8.6</td>
</tr>
<tr>
<td>Equivalent weight</td>
<td>1105.7</td>
<td>1018.3</td>
<td>1173.7</td>
<td>1018.3</td>
<td>920.8</td>
<td>1139.0</td>
<td>1018.2</td>
<td>1060.6</td>
</tr>
<tr>
<td>Ash—%</td>
<td>1.57</td>
<td>--</td>
<td>1.43</td>
<td>--</td>
<td>1.26</td>
<td>--</td>
<td>1.85</td>
<td>1.53</td>
</tr>
<tr>
<td>Methoxyl—%</td>
<td>10.61</td>
<td>12.01</td>
<td>9.97</td>
<td>9.93</td>
<td>9.88</td>
<td>10.61</td>
<td>10.16</td>
<td>10.50</td>
</tr>
</tbody>
</table>

1. Anhydrogalacturonic acid in alcohol precipitate.
2. Calculated on AGA basis.

equivalent weight. This is a reliable means of determining free acid groups and also a means of measuring degree of methylation on a purified sample with correction.

The average values of the equivalent weights of pectin reported in this paper extracted from peel, membrane, and juice sacs are 1020, 1077, and 1061, respectively. These results are higher than those reported by Kertesz (5).

**Jelly Grade.**—This term designates the parts of sugar which one part of solid pectin or pectin extract is capable of supporting under prescribed conditions (2, 5). It is the jelly grade of pectin which determines the commercial value of the product; and the greater the degree of polymerization of the polygalacturonic acid, the greater this value. Data on pectin grades in Tables 1, 2, and 3, were calculated from the jelly breaking strength of the jellies measured at 50 cm. of water pressure. The average values of the pectin grades during the growing season were 206, 314, and 222, respectively, for peel, membrane, and juice sacs. Pectins from the extracted membrane resulted in the highest grades during the season ranging from 286 to 363. Lowest jelly grades were obtained from the peel, ranging from 190 to 219. These grades reflect the values of both water- and acid-extracted pectins which are present when extracting the various AIS components. By first removing the water-soluble pectin, pectins extracted from protopectin would be of greater jelly grade. This would account for so little variation in the pectin properties reported here during maturation of the fruit. However, this potential mixture is what exists for the processor of citrus products, particularly in the pulp which consists mainly of juice sacs. For the pectin manufacturer, the water-soluble pectin can be leached out with water at room temperature. Water-soluble pectins isolated by the authors (15) from Valencia and Pineapple orange juices had jelly grades of only 89 and 92, respectively.

Pectin from seeds had no jellying power when prepared as 65% sugar jellies. Purity of the extracted seed pectin ranged from 55 to 61% and methoxyl content from 12.1 to 13.6%. These data are not presented in tabular form, but only as information for comparison.

**Jelly Units.**—Since this value is calculated by multiplying yield of pectin by jelly grade, it represents the quantity of jelly that may be produced from a given weight of peel, membrane, or juice sacs. The average values for jelly units obtained during the Valencia orange growth season for peel, membrane, and juice sacs were 51.4, 105.0, and 46.6, respectively. The membrane contained slightly more than twice the jelly units of the other 2 components. The highest jelly units from peel and membrane were obtained in December and from the juice sacs in March.

**Relative Viscosity.**—Viscosities of pectin solutions, unless purified and prepared in more or less
similar manner from the same source, merely indicate the approximate grade of pectin or size of the polygalacturonic chain. Relative viscosities of pectins extracted from membrane were greater than for those pectins extracted from peel and juice sacs. Similarly, the jelly grades of the extracted pectins from membrane were much greater, as previously mentioned. Most polyvalent cations will increase the viscosity of a highly methylated pectin; however, due to the use of Zeo-Karb most of these cations have been removed.

Ash Content.—The percentages of ash in the pectins, extracted from each of the 3 component parts during the months of December, February, April, and June, were determined. The results are reported in Tables 1, 2, and 3. Ash indicates the inorganic impurity in the pectin that is probably combined with the carboxyl groups to form pectinic acid salts. A small part of the ash, mainly silica, is occluded in the alcohol precipitate and not combined. Further analysis of the ash contents to determine the metallic ions present in these pectins will be reported later.

SUMMARY

Evaluations were made of pectins extracted from peel, membrane, and juice sacs of Valencia oranges during maturation of the fruit. The pectins extracted from membrane were highest in yield, jelly grade, purity, relative viscosity, and generally higher in degree of methylation. Pectins extracted from the juice sacs were lowest in purity and yield but higher in jelly grade and slightly greater in degree of methylation than those pectins from peel.

Pectins extracted from the seeds had no jelly grade.

The order of component parts for jelly units in the Valencia orange from highest to lowest was found to be membrane, peel, and juice sacs. Peel was second in order because of the percentage yield of pectin and not because of jelly grade, which was greater in the pectin from juice sacs.

LITERATURE CITED


THE EFFECTS OF STORAGE CONDITIONS ON SUGAR CONTENT OF FRESH BLACKEYE PEAS

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Florida Agricultural Experiment Station
Gainesville

Considerable interest in fresh-marketed and processed Blackeye peas, Vigna sinensis (L.) Savii, exists in the southeastern states. Kuhn (6) reported that deterioration of this vegetable after harvest limits its acceptable fresh shelf-life to approximately 10 days. He found that subjective organoleptic evaluations revealed noticeable alterations in both the lipid and carbohydrate components. The quality of shelled peas deteriorated more rapidly than that of unshelled. Kertesz (5) reported rapid decreases of total sugars in English peas after harvest. Sugar losses were temperature-dependent and amounted...