Correct choice was made only 7% of the time. This shows that the microwave treatment does not cause a change in flavor at the 99.9% significance level.

Comparison of Microwave vs. Heat Processing

Commercial Valencia orange juice (30 gallons) was obtained and divided into two lots. One lot was treated in the microwave at 88-91°C with 15 seconds residence time. The second lot was treated in a pilot plant plate pasteurizer at 91°C at 0.75 gal/min.

Pectinmethylesterase and bacteria levels were measured and taste was evaluated by comparing the heat pasteurized vs. the microwave treated vs. the control using the triangle test previously described. Table 3 shows the results. It appears that there is some post pasteurization contamination in the steam heat pasteurizer for bacteria counts. Taste evaluation showed no difference in taste between any of the processes tested (data not shown).

Table 3. Bacteria and PME comparison of control, heat pasteurizer, and microwave pasteurizer.

<table>
<thead>
<tr>
<th>Process</th>
<th>Temperature (°C)</th>
<th>% PME inactivation</th>
<th>CFU/ml¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microwave Pasteurizer</td>
<td>88-91</td>
<td>&gt; 99</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>Steam Heat Pasteurizer</td>
<td>91</td>
<td>&gt; 99</td>
<td>5 x 10⁴</td>
</tr>
<tr>
<td>Control (Fresh)</td>
<td>—</td>
<td>(43 PME units)</td>
<td>2 x 10⁴</td>
</tr>
</tbody>
</table>

¹CFU: Colony Forming Units.

Conclusion

Our microwave continuous flow pasteurizer operated smoothly and gave 37% efficiency of conversion of electrical to thermal energy. Treatment with microwave energy at time/temperature values comparable to commercial pasteurization in metal equipment gave excellent control of bacteria and pectinmethylesterase. There was no adverse effect on taste by microwave treatment.

Literature Cited


ANALYSIS OF ORANGE JUICE VOLATILES: COMPARISON OF EXTRACTION WITH FREON 113 AND ETHYL ACETATE

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Additional index words. flavor degradation products.

Abstract. Much of the objective analyses of citrus juice flavor compounds is performed by gas chromatography. This analytical method is limited to the volatile fractions. This paper deals with solvent extraction of citrus juice using Freon 113 and ethyl acetate solvents. The effects of solvent quantity, temperature, time, mixing methods and phase separation techniques have a direct bearing on whether a particular solvent system is successful. Reproducibility is a vital factor in choosing a solvent extraction system. The technique should provide repeatable results under varying conditions and with different analysts. Freon 113 exhibits better performance with respect to flavor degradation components and reproducibility. Ethyl acetate performs well for analysis of the more volatile flavor compounds but exhibits poor concentration capabilities.

Ethyl acetate is a strong solvent for extracting important flavor volatiles from citrus juice. Some disadvantages of ethyl acetate are: 1) it is easily bound by the pectin and pulp resulting in low recovery of the solvent, 2) it requires relatively high vacuum and temperature to concentrate, resulting in excessive component degradation and unequal loss of volatiles. Freon 113 is a moderate polarity solvent, but due to its refrigeration properties, it can be easily concentrated to as much as 50:1 with minimal heat and a low vacuum. This minimizes degradation and unequal losses of the various recovered components. Release of Freon to the atmosphere is heavily regulated when used for refrigeration and air conditioning but is exempt from such regulation when used for research (U.S. Environmental Protection Agency, 1992).

Materials and Methods

Commercial orange juice was used for this series of experiments. Several containers of juice were mixed together and made up to 2L to eliminate any variation due to different containers. Gas chromatographic analysis was performed with a Hewlett Packard 5890 equipped with a split injector and FID detector. The column used was a 30M Restek RTX-5, (.32 mm diam., .5 μ film). The carrier gas was hydrogen and all injections were .4 μl with a 50:1...
split ratio. Temperature programming was 30°C for 3 min, ramp at 7.5°C/min, and final temperature of 230°C.

Results and Discussion

The first parameter investigated was solvent recovery. Normally, high solvent recovery leads to high analyte recovery. In the case of Freon 113, high solvent recovery is accomplished by having a large interface area between the solvent and the juice. This is normally accomplished by placing the two liquids in a large diameter flask. Slow stirring is used to accelerate exchange of compounds at the boundary layer (Brael, 1984). Over 85% recovery of the solvent is normal with this method when used on citrus juices, but recovery of analytes is poor. When Freon 113 and juice are highly agitated, recovery of solvent drops to 45-50%, but the recovery of analytes is very high. Also, processing time is greatly reduced.

The system selected for both Freon 113 and ethyl acetate extraction utilized the MIXXOR (Cole-Parmer Instrument Co., Chicago, IL). The Mixxor system consists of a graduated mixing reservoir into which a mixer-separator piston fits snugly. The piston has an axial bore leading to a stoppered collecting chamber. In-and-out motion of the piston causes the two liquids to homogenize in the axial bore. This process is followed by centrifugation to promote and accelerate separation. Optimal analyte recovery occurred with agitation at 1 stroke every 5 sec for 3 to 5 min followed by centrifugation at 1,000 G for 15 min. Using this procedure, no difference was found in analyte recovery over a range of juice temperatures of 0 to 28°C for both Freon 113 and ethyl acetate. Ethyl acetate mixes more readily with orange juice than Freon 113, but is lost within a gelatinous mixture layer of pectin and cloud. This results in greater loss and lower recovery. The solvent/juice ratio for ethyl acetate should be at least 1:1, and for Freon 113 a ratio of 1:2 is adequate.

Concentration techniques were investigated next. The refrigeration properties of Freon 113 made it the easiest to concentrate since it vaporizes at moderate vacuum and temperature. A Buchi Rotavapor-R from Brinkman Scientific was used for the vacuum concentration process. The Freon 113 extract was kept at 30°C with a water bath to counteract the chilling effect of vaporization. The vacuum was varied between 280 and 560 mm Hg. Low vacuum resulted in excessive concentration times. High vacuum resulted in excessive loss of the more volatile fractions. The ideal vacuum was a range of 350-400 mm Hg. A 4 ml extract of Freon 113 could be concentrated to 0.08 ml in about 5 min with minimum loss of the most volatile fractions. The ratio of peak areas was nearly constant over a concentration range of 20 to 40:1, thus simplifying quantitative calculations. Ethyl acetate is more difficult to concentrate and still retain the volatile compounds. The water bath temperature was elevated to 40°C and vacuum raised to 650 mm; it took 10 min to concentrate to 4:1. At these higher settings, volatile components were lost at disproportionate rates, which prevents accurate quantitation of the volatiles. Time under vacuum during the concentration step was the most critical factor in obtaining reproducible results. In an extraction system, ethyl acetate is best utilized without concentration.

Figure 1 is a typical gas chromatogram of a Freon 113 extract of orange juice concentrated 40:1. Figure 2 is a typical gas chromatogram of an ethyl acetate extract of the same juice, but not concentrated. In comparison, it can be seen that the most volatile compounds (t/rt<7 min) are extracted more efficiently by ethyl acetate. Freon 113 is more effective with the monoterpenes and aldehyde region (t/rt 10-20 min) than ethyl acetate. This is an important area when studying degradation of aged juices. Tests run with orange juice spiked with furfural indicated a detection limit of 5 ppb and linear recovery over a range of 10 to 100 ppb. Previous attempts at head space analysis for furfural were unsuccessful due to poor sensitivity. Freon 113 extraction provides a convenient tool for monitoring this degradation product.

Figure 2. Gas chromatogram of commercial orange juice extracted with ethyl acetate.

Conclusion

Ethyl acetate is a strong solvent for extraction of citrus juice volatiles but should be used without concentration. Freon 113 is not as strong a solvent but exhibits excellent concentration characteristics that are needed for extracting volatiles from citrus juices, especially degradation products.

The following procedure is recommended for Freon 113 extraction of volatiles from citrus juice:

1. Add 5 ml Freon 113 to 10 ml juice.
2. Mix thoroughly using a MIXXOR, agitating for 5 min.
3. Centrifuge mixture for 15 to 30 min at 1,000 G.
4. Pipet clear liquid into a 10 ml pear-shaped flask.
5. Concentrate the sample with a rotary evaporator using the following conditions:
   A. Water bath at 30°C.
   B. Vacuum ca 330-400 mm Hg.
   C. Medium speed rotation (60-100 rpm).
   D. Condenser coil at 0°C.
6. Do not exceed 50:1 concentration.

**Literature Cited**


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**CONDITION AND QUALITY OF FLORIDA GRAPEFRUIT IMPORTED INTO THE NETHERLANDS DURING 1991-92 SEASON**

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Additional index words. *Citrus paradisi* Macf., decay, total soluble solids, acid, juice volume, export.

**Abstract.** 'Ruby Red' grapefruit (*Citrus paradisi* Macf.) were commercially harvested, packed and shipped in 19 van container shipments to Rotterdam, The Netherlands during September 1991 to April 1992. Three boxes of fruit from each of these 19 shipments were evaluated for condition and quality upon arrival, after holding for 1 week at 15.6 or 10°C and after an additional week at 20°C. The shippers supplied information on pre- and postharvest conditions and treatments. Seasonal changes at harvest and transit temperatures had more effect upon grapefruit condition on arrival and after holding than other variables. Early season fruit which was degreened had more aging and scald and lower rating for appearance and color than mid or late season fruit. Early season fruit had lower soluble solids and soluble solids/acid ratio and higher acid than mid or late season fruit. Rain prior to harvest, no prepacking or SOPP treatment, and degreening increased decay compared to no rain prior to harvest, prepacking and SOPP treatment, and no degreening. Transit temperatures below 4.4°C significantly increased pitting compared to higher transit temperatures. On arrival, about 97% of fruit would be considered marketable, and after 2 additional weeks about 93% was marketable.

In 1991, the United States supplied 41 and 46% of grapefruit quantity and value respectively, imported into The Netherlands (Produktchap voor Groenten en Fruit, 1992). About 90% of all grapefruit imported into The Netherlands from the United States was from Florida and 85% was 'Ruby Red'. Previously, Hillebrand et al., 1978 and Hoogendoorn and Miller, 1986, reported on quality differences of Florida grapefruit compared with other imported fruit. Many studies (Harding and Fisher, 1945; Long et al., 1959, 1960; and Rygg and Getty, 1955) have previously described seasonal changes of grapefruit grown in Florida, Arizona and California. Chace et al., 1966, described the factors affecting the condition of Florida grapefruit exported to Europe upon arrival and after storage. Many studies have been conducted on treating, packaging and shipping Florida grapefruit, both domestically and export, to provide useful marketing information.

The purpose of this study was to determine the condition and quality of 'Ruby Red' grapefruit imported to Europe from Florida and relate the findings to various pre- and postharvest conditions and treatments. The study was conducted on commercially grown, packed and shipped grapefruit.

**Materials and Methods**

A total of 19 shipments from four shippers were made of 'Ruby Red' grapefruit harvested between September 19, 1991 and April 2, 1992 and shipped to Rotterdam, The Netherlands by refrigerated van containers. Total elapsed time from harvest to arrival and first inspection was between 18 and 24 days for degreened fruit and 15 and 22 days for nondegreened fruit. Shippers of the grapefruit supplied information on harvesting, degreening, packing and shipping dates, pre- and postharvest treatments and environmental conditions. Degreening time ranged from 48 to 84 hours, temperature from 26.7°C to 29.4°C and relative humidity from 96 to 100%.

On arrival at the importer's warehouse, three boxes from each shipment were retrieved. The temperature settings of the van containers and actual transit temperatures were verified. The boxes of fruit were taken to the USDA laboratory in Rotterdam for storage and evaluation.

At the laboratory, the initial inspection was conducted within four hours of arrival. After the initial inspection, all the fruit was placed in storage for one week at either 15.6°C or 10°C. Early season fruit harvested before January 1 was stored for four weeks. Fruit from each ship was kept under two different storage temperatures to test the effect of storage temperature on fruit quality.

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