evaluated during the 1985-86 through 1989-90 seasons for limonin content using enzyme immunoassay methodology. The lowest and most constant levels of limonin on a seasonal basis were found in GJFC packed in containers other than cans. Graphic representations of limonin content on monthly basis show the ability of the Florida processors to package grapefruit juice containing generally constant limonin levels. This was especially noticeable in the graph depicting monthly levels in reconstituted grapefruit juice not packed in cans.

Literature Cited


A MICROWAVE SYSTEM FOR CONTINUOUS PASTEURIZATION OF ORANGE JUICE

Seifollah Nikdel and Donald G. MacKellar
Florida Department of Citrus
Scientific Research Department
Citrus Research and Education Center
700 Experiment Station Road
Lake Alfred, FL 33850

Abstract. A continuous pasteurization system was designed based on a domestic microwave oven. Juice was pumped through coils of glass or teflon tubing placed in the oven cavity. Heating characteristics of orange juice were similar to those of water. Residence times were varied by using heating tubes of varying length. A counter current heat exchanger was used to preheat incoming juice and to cool the treated juice. Juice temperature was controlled by varying the flow rate at 100% microwave power. Efficiency was good, with over 90% of the microwave energy absorbed by the juice. Taste tests showed no difference between controls and juice treated at 95°C. Pectinmethylsterase (PME) activity was reduced to <.1% at temperatures of 75° to 95°C for residence times of 15 to 25 seconds.

Citrus products are usually pasteurized to reduce the number of viable bacteria and to inactivate pectinmethylsterase. This enzyme causes loss of product quality during storage. Pasteurized juice has a longer shelf life and can be adapted to aseptic packaging. Pasteurization is usually accomplished by passing the product through a metal heat exchanger, either shell and tube or plate type. The process is time and temperature dependent as described by Eagerman and Rouse (1976). Typical conditions are 90°C for 10 to 60 seconds.

Treatment of citrus juice requires care to prevent development of off flavors. Heat transfer to fluids from a metal surface results in a thin layer of low velocity fluid adjacent to the metal surface. This layer is hotter than the bulk of the juice and it is here that thermal degradation of the juice can take place. To minimize this degradation, flow rates are kept high to provide turbulent flow and the temperature differential is kept low by using hot water or low pressure steam as the heating medium.

In contrast, microwave energy does not present this problem. The radiant energy heats the juice directly without heating the tube walls. There are no heat transfer films to worry about and temperature control is improved. Rapid start up and shut down are possible and less heat is lost to the environment. Microwave heating of foods is frequently done on a commercial basis and is not new for orange juice. Copson (1954) used it, but his device was cumbersome and would be difficult to scale up. He did achieve good control of PME activity in orange juice using 2450 MHz (million cycles per second) frequency. Ericson (1975) described the “Sargent Electronic Evaporator” which had limited commercial use. He used a lower frequency, 30 MHz, to provide heat for evaporation rather than for pasteurization. There are two excellent reviews of general microwave technology which briefly mention juice processing. These are Decareau (1985) and Mudget (1982).

Pasteurization of milk with microwave energy has also been studied. Good results were obtained by Hamid (1969) using crude equipment. Recently, a paper by Kudra et al. (1991) reported on microwave pasteurization of milk. The apparatus these authors used is very similar to the one used in the present study on orange juice. The purpose of this study was to evaluate the suitability of microwave technology for orange juice pasteurization.

Materials and Methods
A domestic microwave oven (GE#JE1019H) was modified for continuous flow treatment of citrus juice. The mode stirrer was converted to variable speed air drive. Two bulkhead fittings for 1/4” tubing were installed in the lower left side of the cavity. These fittings would accept a variety of 1/4” coils of glass or Teflon tubing. The fittings were connected outside the cavity to 1/4” stainless steel 90°
fitting to prevent microwave leakage from the cavity. These fittings were immediately followed by teflon adapters for stainless steel thermocouples, one for entering and one for exiting fluid. The exiting fluid entered a heavily immersed in a refrigerated bath. The length of the coil inside the chamber could be adjusted by adding or removing sections so as to vary the residence time. Thermocouples were located at the end of the holding tube and at the exit to the cooling coil. Fluid flow was maintained using a magnetically coupled gear pump driven by a variable speed motor (Micro pump 81406). This provided flows of 0-200 ml/min. Flow rate measurement with a variable area flowmeter worked well with water, but solids interfered with juice measurement. The flow system was modified to feed from a closed reservoir with air being allowed to enter the reservoir through a flowmeter. The flowmeter was calibrated by directly measuring the volumes of juice delivered over varying times.

The oven power output was determined by the water load method, which measures the temperature increase of 1 Kg sample of water heated for 2 minutes at a starting temperature of 20°C. Power is equal to $35 \times \Delta T$. This includes the conversion of calories to watts, the specific heat of water, time and mass. The output was 570 watts (w) using a line power of 1400 watts or 41% efficiency. The microwave frequency was 2450 MHz.

The system was sanitized by pumping 200 ppm NaOCl solution through it at 60°-70°C for 5 minutes. This was followed by a rinse with sterile water. The oven was operated at 100% power to avoid the temperature cycles which would result from the intermittent operation of the magnetron at lower power settings. Juice temperature was controlled by adjusting the flow rate using the variable speed pump monitored by the flowmeter. The usual temperature variations during a run were ± 1°C.

Bacteria testing was carried out using orange serum agar incubated at 30°C for 48 hours. Inoculated orange juice for evaluating bacteria control was prepared by inoculating sterile, single strength orange juice with Lactobacillus plantarum, culturing it overnight and harvesting the cells. The cells were resuspended in fresh orange juice to eliminate the gas formed during bacterial propagation.

Pectinmethylesterase activity was determined by measuring the amount of acid liberated by PME catalytic hydrolysis of pectin at 30°C when the sample is maintained at pH 7. The activity is reported as PME units.

$$\text{PME} = \frac{\text{m mole acid released}}{\text{ml sample} \times \text{time in minutes}} \times 10^4$$

**Results and Discussion**

**Heating Rate**

The heating rate was measured by pumping water or orange juice at varying rates through the system. The microwave was operated at 100% power, and fluid starting temperature was 26°C. The results are shown in Table 1. The temperature and flow rates in Table 1 can be used to calculate that 90% of the 570 watts microwave power is converted to heat in the water and orange juice. Because the magnetron is 41% efficient, this results in a 37% overall electric efficiency. Comparison of the values for water and orange juice shows that flow rates are nearly equal based on the water content. The sugars present in the orange juice do not contribute significantly to the heating rate.

**Sterilization and Pasteurization**

Nikdel et al. (1992) previously reported that orange serum inoculated with Lactobacillus plantarum required a 20 second residence time at 70°C to achieve a 99.8% reduction in bacteria count. A further experiment was conducted to evaluate both bacteria control and PME inactivation. Hamlin orange juice was treated in the microwave unit at two temperatures, 70°C and 90°C. The unit was set up so that different lengths of 1/4" teflon coil could be used. The different flow rates needed to maintain the two desired temperatures resulted in varying residence time in the chamber. Samples were drawn after equilibrium was obtained at each temperature and tested for bacteria count and pectinmethylesterase activity. The results are shown in Table 2.

This data shows that bacteria are readily killed at either 70°C or 90°C at very short residence time. Pectinmethylesterase (PME) is more resistant and control was not complete at 70°C even at 26 second residence time. PME was inactivated readily at 90°C.

**Flavor Evaluation**

One gallon of Valencia orange juice was treated at 89-94°C with a 15 second residence time. PME activity was reduced 99.7%, and bacteria were reduced to < 20 colony forming units/ml (CFU/ml). The juice was evaluated for flavor change using the triangle taste test. In the test, sets of 3 samples are tasted. The sets contain randomly 2 of 1 juice and one of another. The taste panelist is asked to determine which 2 of the three are identical. Pure chance would result in 33.3% correct answers. Significantly higher or lower values would indicate that there is either a difference or no difference between the samples tested. The treated juice was compared to the untreated juice and the

<table>
<thead>
<tr>
<th>Flow rate (ml/sec)</th>
<th>Equilibrium temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>water</td>
</tr>
<tr>
<td>1.74</td>
<td>95</td>
</tr>
<tr>
<td>1.88</td>
<td>90</td>
</tr>
<tr>
<td>2.04</td>
<td>85</td>
</tr>
<tr>
<td>2.22</td>
<td>80</td>
</tr>
<tr>
<td>2.72</td>
<td>70</td>
</tr>
<tr>
<td>3.49</td>
<td>50</td>
</tr>
</tbody>
</table>

**Table 1. Flow rate vs. water and orange juice temperature.**

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Residence time (sec)</th>
<th>PME inactivation (%)</th>
<th>CFU/ml×*</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>control</td>
<td>22.0</td>
<td>1.79 × 10³</td>
</tr>
<tr>
<td>70</td>
<td>12</td>
<td>86.3</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>70</td>
<td>18</td>
<td>93.2</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>70</td>
<td>26</td>
<td>97.8</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>90</td>
<td>18</td>
<td>99.5</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>90</td>
<td>28</td>
<td>100</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>90</td>
<td>41</td>
<td>100</td>
<td>&lt; 20</td>
</tr>
</tbody>
</table>

*CFU: Colony Forming Units.

**Table 2. PME activity and bacteria count vs. residence time.**

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>
<thead>
<tr>
<th>Process</th>
<th>Temperature (°C)</th>
<th>% PME inactivation</th>
<th>CFU/ml&lt;sup&gt;6&lt;/sup&gt;</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 × 10&lt;sup&gt;5&lt;/sup&gt;</td>
</tr>
<tr>
<td>Control (Fresh)</td>
<td>—</td>
<td>(43 PME units)</td>
<td>2 × 10&lt;sup&gt;5&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>6</sup>CFU: 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

M. Klim and S. Nagy
Florida Department of Citrus
700 Experiment Station Road
Lake Alfred, FL 33850

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