EVALUATION OF A BEVERAGE CLOUDING AGENT FROM ORANGE PECTIN POMACE LEACH WATER

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Abstract. A beverage clouding agent was manufactured from orange pectin pomace leach water prepared from Valencia and Hamlin oranges. A commercial pectinase was used to hydrolyze pectin in the leach water. Clouding agent solids recovered (as % of peel solids) were 29% for Valencia orange peel and 27% for Hamlin orange peel. The stability of the prepared cloud was evaluated at a solids level of 1%. The initial cloud value was 410 NTU and after 30 days storage the drink retained 90% of the initial cloud. Sensory evaluation of the clouding agent indicated no bitterness when 0.25, 0.50, or 1.0% of the clouding agent was added to citrus drinks. Browning of the cloud concentrate during storage was prevented by freezing or by treatment with sulfur dioxide.

Various material are used in most fruit-base drinks to provide an opaque or cloudy appearance. These materials are referred to as “clouding agents”. The major use of these agents is in beverages to which citrus oil-flavoring materials have been added. The citrus oil is emulsified in the aqueous phase with the aid of emulsifiers and stabilizers, as brominated vegetable oil (BVO, sp. gr. 1.2 to 1.3) and glycerol ester of wood rosin (EWR, sp. gr. 1.1)) are added to the citrus oils. The specific gravity of citrus drinks is 1.04 - 1.05. These materials are approved for use by the Food and Drug Administration at a maximum level of 15 ppm to hydrolyze pectin in the leach water. Clouding agent solids recovered (as % of peel solids) were 29% for Valencia orange peel and 27% for Hamlin orange peel. The stability of the prepared cloud was evaluated at a solids level of 1%. The initial cloud value was 410 NTU and after 30 days storage the drink retained 90% of the initial cloud. Sensory evaluation of the clouding agent indicated no bitterness when 0.25, 0.50, or 1.0% of the clouding agent was added to citrus drinks. Browning of the cloud concentrate during storage was prevented by freezing or by treatment with sulfur dioxide.

The production of this new specialty is also desirable because it helps eliminate one source of water pollution during pectin manufacturing. Citrus peel must be leached with water to remove some of the sugars prior to drying the peel or extracting the pectin. Additional information on pectin manufacturing can be found in (1 & 4). Water from the peel leaching operation is concentrated so both the volume and pollution load can be reduced.

The purpose of this study was to evaluate the preparation of a beverage clouding agent prepared from the peel of Florida grown oranges. Emphasis was on the determination of product yield, product composition and physical characteristics of the clouding agent.

Materials and Methods

Clouding Agent Preparation. Orange peel used to prepare the clouding agent was from Valencia oranges picked July 25, 1978 and from Hamlin oranges that were picked November 29, 1978. The fruit was obtained and clouding agents were prepared at the University of Florida Agricultural Research and Education Center at Lake Alfred, Florida. The preparation of the orange peel clouding agent followed the outline presented in Figure 1.

Oranges were washed the day before the clouding agent preparation and stored at ambient temperature overnight. The following morning, the juice was extracted at 2.1 kg/cm² (30 psi) in a FMC (Food Machinery Corporation) in-line juice extractor. Peel was comminuted in a Fitzmill disintegrator type D using 1.15 cm (.020") screen.

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Up to this point the peel has been handled as it would be in a pectin operation. Normally room temperature water is used to remove the soluble solids prior to drying the peel or for extracting the pectin. See (4) for further details.

Ground peel was then added into water which had been preheated to 70°C in a steam jacketed kettle (2.5 kg of water to 1 kg of ground peel). After heating for 5 minutes at 60°C, the mixture was pumped through an FMC finisher model 35 with 2.9 kg/cm² (42 psi) air pressure, and a .05 cm (.020") screen.

Peel that has been leached with hot water has been previously examined and found suitable for extracting pectin (5, 6, 7, 8).

Liquid recovered from the finisher was pumped through a SA7-06-076 Centrico continuous centrifuge which removed large sludge particles from the liquid extract. The centrifuged extract was enzyme treated with a pectinase (Irgazyme 100, CIBA Geigy Labs) for 30 minutes at 52-54°C. Enzyme concentration was 100 ppm (by weight) for Valencia orange

peel and 200 ppm for Hamlin orange peel. After 30 minutes at 52-54°C the enzyme was inactivated by heating to 83°C for 3 minutes. The enzyme treated liquid extract was designated as a single strength clouding agent. A portion of this single strength clouding agent was concentrated in a PS (Precision Scientific Co.) vacuum glass evaporator at 50°C to a Brix of 48.9 for Valencia orange peel clouding agent and 51.4 for Hamlin orange peel clouding agent.

Table 1. Material balance for the cloud extraction process.

<table>
<thead>
<tr>
<th></th>
<th>Pounds</th>
<th>Percent solids</th>
<th>Pounds</th>
<th>Yield, % of peel solids</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Val</td>
<td>Ham</td>
<td>Val</td>
<td>Val</td>
</tr>
<tr>
<td>Fruit</td>
<td>191</td>
<td>292</td>
<td>25.6</td>
<td>21.7</td>
</tr>
<tr>
<td>Ground peel</td>
<td>77</td>
<td>120</td>
<td>14.1</td>
<td>10.2</td>
</tr>
<tr>
<td>Finisher discharge</td>
<td>81</td>
<td>158</td>
<td>14.1</td>
<td>10.2</td>
</tr>
<tr>
<td>Centrifuge discharge</td>
<td>22</td>
<td>24</td>
<td>9.3</td>
<td>8.4</td>
</tr>
<tr>
<td>Cloud extract</td>
<td>182</td>
<td>200</td>
<td>3.1</td>
<td>3.5</td>
</tr>
</tbody>
</table>

ORANGE PEEL CLOUDING AGENT.

PROCESS DIAGRAM.

FRUIT -> JUICE

PEEL

SHRED PEEL

WATER MIXER - 60°C

FINISHER PECTIN POMACE

CENTRIFUGE

ENZYME TREATMENT 52 - 54°C

ENZYME INACTIVATION 83°C

CENTRIFUGE

CLOUD EXTRACT

VACUUM CONCENTRATION 50°C

WATER VAPOR

CLoudING AGENT

Fig. 1. Schematic of the process for manufacture of citrus clouding agent.

Brix, Titratable Acidity, and Ascorbic Acid. Determined by standard industry procedure (10).

Total Solids. Percentage of total solids was evaluated by drying for twelve hours in a vacuum oven at 70°C and 25 mm of mercury. Samples were cooled in a desiccator and weighed to obtain percent total solids.

Pectins. Water-soluble, ammonium oxalate, and sodium hydroxide pectin fractions were evaluated by the method described by Rouse and Atkins (12).

Reducing and Invert Sugars. Sugars were determined by a colorimetric method described by Ting (15).

Relative Viscosity. Viscosity was measured with a Brookfield Viscometer Model LVT at 25°C and 30 rpm with spindle #4.

Recoverable Oils. Recoverable oils were measured by the Scott procedure (10).

Turbidity. A Hach Model 2100A Turbidimeter was used to evaluate samples which had been diluted to 1 percent solids. Latex solution standards were used for instrument calibration.

Spectrophotometric Determination of Browning. The procedure of Meydev et al. (11) was used. The absorbance of ethanol extracts of the cloud concentrate was measured at 490 nm.

Results and Discussion

A material balance for the cloud preparation process is presented in Table 1. The process resulted in a yield of clouding agent per 40.9 kg (90 lb) box of fruit of 2.3 lbs solids for Valencia oranges and 2.1 lbs. solids for Hamlin oranges. Total solids content of the single strength beverage clouding agent ranged from 3.2 to 3.5 percent (Table 2). The reducing sugars accounted for 55 to 61 percent of the total sugars. Total sugars, as invert, were 67 to 74 percent of the total solids. The water soluble pectin fraction was 77 percent of the total pectin and the ammonium oxalate and sodium hydroxide soluble fractions each accounted for approximately 12 percent of the total pectin. The high value for the water soluble pectin would be expected with the warm water extraction procedure. The concentrate had a viscosity of 9200 to 9800 cps, which is extremely viscous. A standard value for 45° Brix FCOJ would be in the range of 500 to 1000 cps. In a later experiment the pectinase enzyme concentration was increased to 500 ppm and the viscosity of the 50° Brix cloud concentrate produced was 500 cps. The color stability of the concentrated cloud extract was good when stored at either 2°C or −26°C for 60 days (Fig. 2). There was significant browning of the concentrate at 18°C and 10°C. The addition of 2000 ppm sodium metabisulfite controlled the browning at all four storage temperatures. The sulfur dioxide treated cloud concentrate after 60 days of storage was lighter in color than the original unstored concentrate. The addition of 1.0 percent clouding agent solids to a citrus drink base produced a cloud value of 410 nephelometric turbidity units (NTU). After 30
Table 2. Properties of Valencia and Hamlin orange peel single strength and concentrated clouding agents.

<table>
<thead>
<tr>
<th>Property</th>
<th>Valencia orange&lt;sup&gt;SS&lt;/sup&gt;</th>
<th>Concentration</th>
<th>Hamlin orange&lt;sup&gt;SS&lt;/sup&gt;</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soluble solids (&lt;sup&gt;°&lt;/sup&gt;Brix)</td>
<td>3.1</td>
<td>48.9</td>
<td>3.5</td>
<td>51.4</td>
</tr>
<tr>
<td>Total solids (%)</td>
<td>3.2</td>
<td>50.9</td>
<td>3.4</td>
<td>53.0</td>
</tr>
<tr>
<td>Titratable acidity (% citrate)</td>
<td>0.7</td>
<td>0.95</td>
<td>0.7</td>
<td>1.0</td>
</tr>
<tr>
<td>Essential oil (%)</td>
<td>0.114</td>
<td>—</td>
<td>0.142</td>
<td>—</td>
</tr>
<tr>
<td>Water soluble pectin (% AGA)</td>
<td>0.195</td>
<td>—</td>
<td>0.199</td>
<td>—</td>
</tr>
<tr>
<td>Ammonium oxalate pectin (% AGA)</td>
<td>0.032</td>
<td>—</td>
<td>0.085</td>
<td>—</td>
</tr>
<tr>
<td>Sodium hydroxide pectin (% AGA)</td>
<td>0.027</td>
<td>—</td>
<td>0.080</td>
<td>—</td>
</tr>
<tr>
<td>Reducing sugars (g/100 ml)</td>
<td>1.1</td>
<td>22.0</td>
<td>1.6</td>
<td>24.0</td>
</tr>
<tr>
<td>Invert sugars (g/100 ml)</td>
<td>2.1</td>
<td>39.1</td>
<td>2.6</td>
<td>42.2</td>
</tr>
<tr>
<td>Relative viscosity (cp)</td>
<td>—</td>
<td>9260</td>
<td>—</td>
<td>9840</td>
</tr>
<tr>
<td>Ascorbic acid (mg/100 ml)</td>
<td>Traces</td>
<td>Traces</td>
<td>Traces</td>
<td>Traces</td>
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</tbody>
</table>

<sup>SS</sup> = single strength, conc. = concentrate.

days storage the citrus drinks retained 90 percent of the original cloud value. Commercial drinks evaluated for cloud ranged from 60 to 140 NTU.

Previous work (1) had indicated that bitterness was a problem with some citrus clouding agents. The cloud extract was added to a standard drink base at levels of 0.25, 0.50 and 1.0 percent by weight and evaluated by a sensory panel. The panelists detected no difference between the drinks with cloud added and drinks without cloud.

**Conclusion**

1. A citrus cloud was prepared from peel with a yield of 0.95 kg (2.1 lb) and 1.04 kg (2.3 lb) solids per 40.9 kg (90 lb) box of oranges.
2. The cloud preparation could fit into the process for pectin manufacture or a process for the production of fiber from citrus peel.
3. The cloud concentrate may be frozen or treated with sulfur dioxide to prevent browning.
4. The cloud maintained good stability following frozen storage.
5. In a sensory panel evaluation of citrus drink to which cloud concentrate was added at 1 percent, no bitterness was detected.

**Literature Cited**

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Fig. 2. The effect of storage temperature and bisulfite treatment on the color stability of citrus cloud concentrate.

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