Production of high quality citrus pectin peel

Susanne Oxenbøll Sørensen, CP Kelco ICBC, September 17th 2015
In case you are already fantasizing about drinks in the bar…. 

• High quality citrus pectin peel 
  – Efficient washing and drying process 
  • Peel particle size 
  – Control of enzymatic activity 
• Time 
• pH 
  – Don’t burn the pectin peel!!!
• CP Kelco
• Pectin
• Citrus pectin peel
• Parameters affecting peel quality
• Summary
• CP Kelco
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• Summary
• CP Kelco is a hydrocolloids producer 100% owned by the JM Huber Corporation

• JM Huber is a 120 year old family owned corporation comprising CP Kelco, Huber Engineered Woods and Huber Engineered Materials

• CP Kelco’s Hydrocolloid portfolio
  – Pectin
    • Extracted from citrus peel (DEN, GER, BRA)
  – Carrageenan
    • Extracted from seaweed (DEN, PHI)
  – Carboxymethyl Cellulose (CMC)
    • Manufactured from wood pulp or cotton linters (FIN, CHN)
  – Biogums (Gellan, Xanthan and Diutan)
    • Produced by fermentation (USA, CHN)
• **Pectin extraction process**

![Diagram of Pectin Extraction Process]

- **Raw material arriving**
- **Shipping of Pectin**
- **Finished Product**
- **Blending and standardization**
- **Ready for characterization!**
- **Powdered semi-finished product**
- **Milling**
- **Drying**
- **Filtration**
- **Ion-Exchange**
- **Precipitation**
- **Washing**
- **H₂O**
- **Acid**
- **Insoluble Material**
- **IPA 80%**
- **IPA 60%**
Applications – Food & Beverage
- Bakery
- Beverage systems
- Confectionery
- Dairy (cheeses, ice cream, yogurts and desserts)
- Fruit based products including jams/jellies
- Meat, poultry, analogue and fish processing
- Salad dressings, marinades & sauces
- Water dessert gel

Applications – Consumer & Industrial
- Agrochemicals & Animal Feed
- Ceramics
- Construction (viscosity modifying agent) & Paints
- Household Products (paper towels, surface & toilet cleaners, detergents)
- Oral Care
- Personal Care (creams, gels, shampoo)
- Pharmaceuticals
- Textile Printing
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Schematic pectin structure

Rhamnogalacturonan I  Xylogalacturonan  Homogalacturonan  Rhamnogalacturonan II

- = D-Galacturonic acid
- = L-Rhamnose
- = D-Galactose
- = D-Glucuronic acid
- = L-Arabinose
- = L-Galacturonic acid
- = L-Galactose
- = Kdo
- = L-Fucose
- = D-Apiose
- = D-Xylose
- = D-Dha
- = O-Acetyl
- = O-Methyl
- = Borate
• Model of plant cell wall structure - pectin is the glue
• Pectin quality parameters
  – Degree of esterification (DE)
  – Molecular size (Intrinsic viscosity – IV)
  – Galacturonic acid content (GA)
• Degree of methyl esterification
  – DE > 50% (HM)
  – DE < 50% (LM)

Methyl esterification of acid groups affects calcium reactivity

PLANT PHYSIOLOGY, Third Edition, Figure 15.13 (Part 2) © 2002 Sinauer Associates, Inc.
• Molecular size (Intrinsic viscosity)
  – Average molecular size
  – Typical unit is dl/g
  – Correlates to grading strength of many applications
  – HPSEC
    • Viscotek
• Galacturonic acid content
  – The amount of GA residues relative to the total amount of pectin
  – Homogalacturonan
  – Considered the “active” part of pectin
• The best preserved pectin precursor is always the preferred choice
• Citrus peel is by nature an excellent precursor
  – High yield and quality
  – Available in sufficient amount
  – Stable and consistent supply...

However...
attention/effort is required to ensure high peel quality
• CP Kelco
• Pectin structure and localization
• Pectin quality
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Total citrus harvest (2014/15)
Source: USDA /FAS
Total crop 88,473 kMT

74% Fresh fruit
26% processed

What happens to the processed peel?
90% Cattle feed + other
10% Dry peel

Estimated annual dry peel production 115,000 MT
56% Lemon
30% Lime
13% Orange
1% Other

>80% of Lemon/Lime wet peel dry pectin peel
1-2% of Orange wet peel dry pectin peel
Parameters affecting peel quality

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Parameters affecting peel quality

Dry pectin peel process

Fruit

Juice/oil extraction

Milling

Wet peel

Dry peel

Compactor

Cooling

Dry peel with 10% moisture in 50 kg bales

Three pass dryer

Pre-dryer single pass

Dry peel with 10% moisture in 50 kg bales

Pre-Screw press

Final Screw press

Pre-Pressing

2 step drying

Tank 3

Tank 2

Tank 1

3-step counter current washing

The What if...You CAN!™ Company

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• Why is washing and drying required?
  – Increase net pectin content = pectin extraction yield
  – Prevent further degradation = stabilize pectin
    • Long distance transport of peel (South America – Europe)
Dry pectin peel process

Parameters affecting peel quality

**Important**
- Fruit ripeness
- Fruit quality
- Residence time of fruit and peel
- De-oiling
- Size distribution after milling

**Important**
- pH
- Brix
- Temperature
- Residence time of wet peel

**Important**
- Drying temperatures
- Cooling of peel to ambient temperature

- Juice/Oil extraction
- Milling of peel
- Counter current washing with water
- Screw press
- Rotary dryer to reach 10% moisture
- Compacting into 50 kg bales
Parameters affecting peel quality

Fruit ripeness/quality and residence time

Fruit
- Juice/Oil extraction
- Milling of peel

Wet peel
- Counter current washing with water
- Screw press

Dry peel
- Rotary dryer to reach 10% moisture
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Important
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- Fruit quality
- Residence time of fruit and peel
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Parameters affecting peel quality

Best quality at time of juicing... ... starts immediately

**Enzymes**
Endogenous (in the peel) or exogenous (microbial or "process aid")

Enzymatic activity depends on temperature and pH
Impact of fruit ripeness and wet peel residence time on peel quality

<table>
<thead>
<tr>
<th>Quality</th>
<th>Hours’ residence time from juice extraction to washing/drying</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0h</td>
</tr>
<tr>
<td>Production month</td>
<td></td>
</tr>
<tr>
<td>Season start</td>
<td>100%</td>
</tr>
<tr>
<td>1 Month</td>
<td>99%</td>
</tr>
<tr>
<td>2 Month</td>
<td>98%</td>
</tr>
<tr>
<td>3 Month</td>
<td>96%</td>
</tr>
<tr>
<td>4 Month</td>
<td>95%</td>
</tr>
<tr>
<td>5 Month</td>
<td>94%</td>
</tr>
<tr>
<td>6 Month</td>
<td>93%</td>
</tr>
<tr>
<td>7 Month</td>
<td>92%</td>
</tr>
</tbody>
</table>

1.2% loss of functionality per month and 3% per hour
Parameters affecting peel quality

De-oiling

**Fruit**
- Juice/Oil extraction
- Milling of peel

**Wet peel**
- Counter current washing with water
- Screw press

**Dry peel**
- Rotary dryer to reach 10% moisture
- Compacting into 50 kg bales

**Important**
- Fruit ripeness
- Fruit quality
- Residence time of fruit and peel
- De-oiling
- Size distribution after milling
• De-oiling prior to juicing
  – De-oiling by gently puncturing oil cells (Flavedo layer) is an advantage versus rasping technology
  • The hard and water repellant peel surface is opened and permeable to water both during peel washing and during pectin extraction process

Parameters affecting peel quality
Parameters affecting peel quality

**Peel particle size**

**Fruit**
- Juice/Oil extraction
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**Important**
- Fruit ripeness
- Fruit quality
- Residence time of fruit and peel
- De-oiling
- Size distribution after milling
Parameters affecting peel quality

• Peel Particle size
  – Uniform particle size is recommended
    • Preferably ~5mm
    • Important to adjust mill according to peel hardness and keep knives sharp
  – Uniform particle size distribution means
    • Easier removal of soluble solids during washing (increasing pectin yield)
    • Gentle but effective drying without burning small particles
      – Larger particles contain more moisture – more difficult to dry
Parameters affecting peel quality

Washing conditions

Fruit
- Juice/Oil extraction
- Milling of peel

Wet peel
- Counter current washing with water
- Screw press

Dry peel
- Rotary dryer to reach 10% moisture
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Important
- pH
- Brix
- Temperature
- Residence time of wet peel
• Washing conditions
  – pH - the "regulator" of enzymes
    • General conditions – we recommend pH 4 (last wash/final press)
      – At pH >4 DE reducing enzymes are active 😞
      – At pH <4 IV reducing enzymes are active 😞
  – Brix – indicator of washing efficiency (removed soluble solids)
    • As low as possible is preferred
    • Depend on local conditions
  – Temperature – ambient is preferred
    • Increased solubility and thus higher washing efficiency at higher temp.
    • But enzymatic activity is then also increased
  – Time – no residence time of washed peel
    • Increased risk of enzymatic degradation
Parameters affecting peel quality

Drying temperatures

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Important
- Drying temperatures
- Cooling of peel to ambient temperature
Parameters affecting peel quality

- **Drying temperature**
  - Pectin chain (IV) degradation accelerates when peel temperature exceeds 90 °C

- **Recommended drying conditions**
  - Air inlet of dryer <500 °C and outlet 110-120 °C
  - Peel outlet of dryer 70-80 °C
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    • Peel particle size
    – Control of enzymatic activity
    • Time
    • pH
  – Don’t ”burn” the pectin peel!!!
Acknowledgements

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