Use of Microcrystalline Waxes in Candles

Presented to ALAFAVE and the NCA by The International Group, Inc (IGI)

June 28, 2007

THE INTERNATIONAL GROUP, INC.
Presentation Overview

- Part I: Chemical Makeup, Manufacturing and Product Origins, Commercial Use and Markets
- Part II: Manufacturing, commercial uses, and market conditions
- Part III: Review of Lab Work and Testing Utilizing Microcrystalline Waxes in Candle Applications
- Part IV: Recommendations
Discussion of Base Properties and Fundamentals

Paraffin compared to microcrystalline wax
Petroleum Wax-Basic Properties

- All petroleum derived waxes are complex mixtures of many different hydrocarbons. Each individual component possesses its own melting point, viscosity, penetration, etc.
- The physical properties of a wax are an average of the physical properties of all of these components
Wax Classifications

- **Paraffin**
  - Low melting
  - White
  - Hard
  - Brittle
  - Translucent
  - Crystalline
  - Glossy

- **Microcrystalline**
  - Higher melting
  - Colored
  - Soft
  - Malleable
  - Opaque
  - Amorphous
  - Adhesive
The simplest paraffin or “alkane” is METHANE with the molecular formula CH$_4$ (1 Carbon, 4 Hydrogen Atoms)
Paraffin, Intermediate & Microcrystalline waxes are all fully saturated hydrocarbon mixtures with the formula:

\[ \text{C}_n\text{H}_{2n+2} \]
Alkanes with 6 - 16 carbon atoms are typically liquids at room temperature - the first real waxy solid is OCTADECANE - C_{18}H_{38}

Note the straight “backbone” - referred to as NORMAL, UNBRANCHED or LINEAR

Melting point - 27C/82F

Typical of PARAFFIN waxes
PARAFFIN MICROPHOTOGRAPH

x200
As the molecular weight increases, there are many more ways to arrange the carbon and hydrogen atoms and still satisfy the bonding requirements of the elements, i.e. Carbon = 4
Hydrogen = 1

Note the complex structure - referred to as branched, non-normal or non-linear.

The 18 carbon atoms and 38 hydrogen atoms can arrange themselves - eg

Typical of **MICROCRYSTALLINE** waxes
MICROCRYSTALLINE PHOTO
X200
# Effect of Branching

<table>
<thead>
<tr>
<th></th>
<th>Melting Point</th>
<th>Viscosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear C24</td>
<td>51.5C / 125F</td>
<td>2.4cps @ 90C</td>
</tr>
<tr>
<td>2 methyl C23</td>
<td>42C / 108F</td>
<td>2.5cps @ 90C</td>
</tr>
<tr>
<td>2,2 dimethyl C22</td>
<td>34.6C / 94F</td>
<td>2.7cps @ 90C</td>
</tr>
</tbody>
</table>

A very minor amount of branching **SIGNIFICANTLY** effects all the physical properties - e.g. melting point, viscosity and penetration (hardness)
GC Profile
Straight Paraffin Wax

Carbon Number

Area

NORMALS
ISOMERS
GC Profile

Microcrystalline Wax

Area

Carbon Number
GC Profile
Paraffin+5% Microcrystalline Wax

Effect of micro wax addition
Microcrystalline Wax
Melt Point versus Molecular Weight

Melt Point

200F
150F
100F

Molecular Weight

400
800

Hardening
Coating
Laminating
In General

**Paraffin Waxes** - mainly linear C18 to C40

**Intermediate Waxes** - Increased branching C25 to C60

**MicroWaxes** - Little or no linear HC
  - Complex, branched structure
  - Many components C25 to C85

The properties of “intermediate” waxes are between those of paraffins & micro’s.
Manufacture of Microcrystalline Waxes

Methods and Processes
Raw Materials Origins

- Microcrystalline waxes are derived from by-products of other petroleum processes.
- Petroleum lubricant manufacture is often the source of these by-products.
- This by-product stream, or “slack wax”, is the residual lubricant stock, or “bottoms” product of the vacuum distillation process used at the oil refinery.
Commercial Use of Microcrystalline Waxes

Industries, Applications, and Market Conditions
# Microcrystalline Wax Classifications

<table>
<thead>
<tr>
<th>Micro Type</th>
<th>Melt Point (°F/°C)</th>
<th>Needle Penetration (dmm)</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laminating Grade</td>
<td>130-170°F / 54.4-76.7°C</td>
<td>20-40</td>
<td>Flexible, Tacky</td>
</tr>
<tr>
<td>Coating Grade</td>
<td>170-185°F / 76.7-85°C</td>
<td>15-25</td>
<td>Harder, Low Tackiness</td>
</tr>
<tr>
<td>Hardening Grade</td>
<td>185-200°F / 85-93.3°C</td>
<td>5-12</td>
<td>Very Hard, Higher Viscosity</td>
</tr>
</tbody>
</table>
# Uses of Micro Grades

<table>
<thead>
<tr>
<th>Laminating Grades</th>
<th>Packing, Adhesives, Cosmetics, Rubber, Candles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating Grades</td>
<td>Adhesives, Packaging, Chewing Gum, Inks, Plastics, Rubber</td>
</tr>
<tr>
<td>Hardening Grades</td>
<td>Adhesives, Inks, Chewing Gum, Candles, Specialty</td>
</tr>
</tbody>
</table>
Market Division of Microcrystalline Waxes

- Packaging: 28%
- Cosmetics/Pharmaceutical: 18%
- Candles: 10%
- Rubber: 7%
- Plastics: 8%
- Adhesives: 10%
- Miscellaneous: 17%
- Chewing Gum: 2%
## Global Microwax Supply

*(Production - MT’s)*

<table>
<thead>
<tr>
<th>Region</th>
<th>Laminating</th>
<th>Coating</th>
<th>Hardening</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>South America</td>
<td>3,000</td>
<td>15,000</td>
<td>0</td>
<td>18,000</td>
</tr>
<tr>
<td>Asia</td>
<td>4,000</td>
<td>25,000</td>
<td>2,000</td>
<td>31,000</td>
</tr>
<tr>
<td>Africa/Middle East</td>
<td>2,000</td>
<td>4,000</td>
<td>0</td>
<td>6,000</td>
</tr>
<tr>
<td>Europe</td>
<td>38,000</td>
<td>25,000</td>
<td>4,000</td>
<td>67,000</td>
</tr>
<tr>
<td>North America</td>
<td>15,000</td>
<td>15,000</td>
<td>6,000</td>
<td>36,000</td>
</tr>
<tr>
<td>Totals</td>
<td>62,000</td>
<td>84,000</td>
<td>12,000</td>
<td>158,000</td>
</tr>
</tbody>
</table>
Using Microcrystalline Waxes in Candle Applications

Benefits and Lab Work
Benefits of Microcrystalline Waxes in Candle Systems

- **Appearance**
  - Increased opacity
  - Aides in obtaining smooth surface, eliminates mottling/crystallization spots
  - In jar candles, can aide and promote adhesion to glass

- **Fragrance Properties**
  - Retention, reduction in fragrance bleed

- **Structural Properties**
  - Strengthens crystal structure, imparted properties on candle molecular structure
  - Increased rigidity and/or flexibility (for wick waxes)
Lab Work Focus on Microcrystalline Waxes

- Reviewed three main applications:
  - Container Candles
  - Pillar/Votive Molded Candles
  - Compression Candles

- Utilized various types and % of micros to examine/quantify the following properties:
  - Opacity
  - Structural Properties
  - Fragrance Bleed
  - Compression Properties
## Microcrystalline Waxes Evaluated

<table>
<thead>
<tr>
<th>Designation</th>
<th>Type</th>
<th>Congeal Point, °F (ASTM D938)</th>
<th>Color (ASTM D6045)</th>
<th>Needle Pen, dmm (ASTM D1321)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Laminating Grade</td>
<td>140</td>
<td>+16</td>
<td>35</td>
</tr>
<tr>
<td>B</td>
<td>Laminating Grade</td>
<td>170</td>
<td>+16</td>
<td>30</td>
</tr>
<tr>
<td>C</td>
<td>Laminating Grade</td>
<td>175</td>
<td>+16</td>
<td>30</td>
</tr>
<tr>
<td>D</td>
<td>Hardening Grade</td>
<td>185</td>
<td>+16</td>
<td>18</td>
</tr>
</tbody>
</table>
Quantifying and Examining Opacity

- Purpose:
  - Demonstrate how the branching effect of a microcrystalline wax will effect the crystalline structure of a paraffin and its opacity
  - Demonstrate the effect microcrystalline wax will have on the opacity of a candle system
  - Allow candle manufacturers to predict and determine how changes to their formula will alter the overall opacity of their color system
Quantifying and Examining Opacity

- Same system as utilized in 2004 NCA presentation
- System setup: Lovibond RT 100 Reflectance Color Measurement System
  - Measurements Done Using the D65 Illuminant
  - Theoretical Average Daylight with a Colour Temperature of approx 6500°K
  - Analysis of Data Designed to Express Results Measured in Co-ordinate Systems as well as Differences
Opacity/Color Measurement Ranges

- Basis: Eye Cone Receptor Information Coded into Light-Dark, Red-Green and Yellow-Blue Signals Before Reaching Brain

- Color Co-ordinates in CIE L*a*b* system:
  - L* - Lightness Co-ordinate
  - a* - Red/Green Co-ordinate
  - b* - Yellow/Blue Co-ordinate
Procedure

- Mid mp paraffin (135 F) blended with the following microcrystalline waxes:
  - 1, 3, and 5% “Micro B” (Laminating Grade)
  - 1, 3, and 5% “Micro D” (Hardening Grade)

- Low melt point scale wax (125 F mp) blended in the following combinations:
  - 1, 3, and 5% “Micro A” (low mp laminating grade)
  - 1, 3, and 5% “Micro B” (laminating grade)
  - 1, 3, and 5% “Micro C” (laminating grade)

- Base paraffin and blend measured on Lovibond RT-100 measurement system
Opacity Metering Equipment

- Lovibond RT100 Reflectance Tintometer
# Measured Opacity Results - Low MP

<table>
<thead>
<tr>
<th>Blend</th>
<th>Average L* Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% 125 mp paraffin</td>
<td>36.08</td>
</tr>
<tr>
<td>+1% Micro A</td>
<td>37.34</td>
</tr>
<tr>
<td>+3% Micro A</td>
<td>38.45</td>
</tr>
<tr>
<td>+5% Micro A</td>
<td>40.31</td>
</tr>
<tr>
<td>+1% Micro B</td>
<td>37.15</td>
</tr>
<tr>
<td>+3% Micro B</td>
<td>38.88</td>
</tr>
<tr>
<td>+5% Micro B</td>
<td>40.43</td>
</tr>
<tr>
<td>+1% Micro C</td>
<td>37.39</td>
</tr>
<tr>
<td>+3% Micro C</td>
<td>38.35</td>
</tr>
<tr>
<td>+5% Micro C</td>
<td>40.24</td>
</tr>
</tbody>
</table>

Reference standard = 94.35

3 to 12% increase

Base Paraffin = 36.08

+1% Micro B = 37.15
+5% Micro B = 40.43

L* = 100
# Measured Opacity Results-Mid MP

<table>
<thead>
<tr>
<th>Blend</th>
<th>Average L* Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% 135 mp paraffin</td>
<td>34.2</td>
</tr>
<tr>
<td>+1% Micro B</td>
<td>37.8</td>
</tr>
<tr>
<td>+3% Micro B</td>
<td>39.8</td>
</tr>
<tr>
<td>+5% Micro B</td>
<td>41.1</td>
</tr>
<tr>
<td>+1% Micro D</td>
<td>36.8</td>
</tr>
<tr>
<td>+3% Micro D</td>
<td>40.1</td>
</tr>
<tr>
<td>+5% Micro D</td>
<td>42.1</td>
</tr>
</tbody>
</table>

7.6 to 23% increase

Base Paraffin=34.2

+1% Micro D=36.8

+5% Micro D=42.1

L*=100
Examining Structural Properties

- **Purpose:**
  - to demonstrate how the addition of microcrystalline wax can benefit the structural integrity of molded candles
  - Microcrystalline wax, by introducing branching to the carbon chain, increases bond strength and thus strengthens the overall structure in poured molded candles
  - The improved strength allows for improved resistance to heat and shipments in hot conditions
Evaluation Procedure

- Candles utilized:
  - Molded votive candles with 130°F mp paraffin wax
  - Molded votive candles modified with 1, 3, and 5% of micro wax “B” (laminating grade)
  - Molded votive candle modified with 1, 3, and 5% of micro wax “D” (hardening grade)
- Candles placed in an oven at 40°C (104°F) for a 24 hr period
- Resulting votive candle visually evaluated for structural defects
Visual Results - Votive Candles

Mid MP Paraffin w/ Micro “B”

5% Micro  3% Micro  1% Micro  No Micro
Visual Results - Votive Candles

Mid MP Paraffin w/ Micro “D”

5% Micro  3% Micro  1% Micro  No Micro
Use of Microcrystalline Wax for Fragrance Retention

- **Purpose:**
  - To demonstrate how microcrystalline wax can inhibit fragrance bleeding

- Introduction of isomers to the candle system results in a smaller crystal structure and thus improved fragrance retention
Evaluation Procedure

- Two systems evaluated
  - Container Candle-low mp paraffin wax vs. low mp paraffin wax blended with micro (A, B, and C)
  - Votive Candle-mid melt point paraffin wax vs. mid melt point paraffin wax blended w/ micro (A, B, and C)
- Each system purposely loaded with a high (10%) fragrance oil
- Container Candles examined visually only for fragrance bleed
- Votive candles weighed before and after fragrance bleed to determine % loss
Container Candle w/ Addition of Micro A
Container Candle w/ Addition of Micro B
Container Candle w/ Addition of Micro C
Fragrance Bleed in Votive Candles

- Mid melt point paraffin combined with 10% fragrance
- Votives made and set for 24 hours. Votives then weighed, de-molded, and cleaned of excess fragrance oil
- Votives re-weighed to determine % fragrance loss
  - Average fragrance loss=20% of added fragrance
  - Micros B, C, and D added at 1, 3, and 5% all stopped the resulting fragrance bleed
Compression Candle Testing

- **Purpose:** to determine the effect micro has on compression candle structure

- **Two subsets examined:**
  - Unscented compressed votives
  - Scented compressed votives

- **Desired Properties from adding micro wax:**
  - Reduce migration of residual oil and/or fragrance oil
  - Reduce cold flow properties
  - Strengthens the candle structure

- **Waxes used**
  - Micro waxes “B” (lam. 170 CP) and “C” (lam. 175 CP)
  - 130°F FRP and 135°F FRP
Equipment and Testing Setup

- Testing performed by SMS Marketing, Inc (Durham, NC)

- Equipment Used
  - Pilot compression machine
  - Imanda, Model PS (resolution +/- 0.2%) compression gauge

- Test Formulations
  - Micro wax at 1% and 2% by weight
  - Fragrance oil at 3% and 5% by weight
  - Votive candles manufactured with pilot compression machine

- Repeatability
  - Each test progression run 12 times
Picture of Pilot Compression Machine-Granulator Drum
Pilot Compression Unit
Compressed Candles
Imanda, Model PS Procedure

- Gauge applied to candle wick hole using bottom probe
- Force applied until candle cracks and breaks
- Gauge “locks on” highest force measurement, in lbs
Compression Testing
## Testing Progression-Votives

<table>
<thead>
<tr>
<th>TEST #</th>
<th>% PAR, type</th>
<th>% MICRO, type</th>
<th>COLOR</th>
<th>% FRG Oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100% 130 mp</td>
<td>0%</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>2</td>
<td>99% 130 mp</td>
<td>1% B</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>3</td>
<td>98% 130 mp</td>
<td>2% B</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>4</td>
<td>96% 130 mp</td>
<td>1% B</td>
<td>Yes</td>
<td>3%</td>
</tr>
<tr>
<td>5</td>
<td>93% 130 mp</td>
<td>2% B</td>
<td>Yes</td>
<td>5%</td>
</tr>
<tr>
<td>6</td>
<td>100% 130 mp</td>
<td>0%</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>7</td>
<td>99% 130 mp</td>
<td>1% C</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>8</td>
<td>98% 130 mp</td>
<td>2% C</td>
<td>No</td>
<td>0%</td>
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<tr>
<td>9</td>
<td>96% 130 mp</td>
<td>1% C</td>
<td>Yes</td>
<td>3%</td>
</tr>
<tr>
<td>10</td>
<td>93% 130 mp</td>
<td>2% C</td>
<td>Yes</td>
<td>5%</td>
</tr>
</tbody>
</table>
# Testing Progression-Votives w/ Mid-MP Wax

<table>
<thead>
<tr>
<th>TEST #</th>
<th>% PAR, type</th>
<th>% MICRO, type</th>
<th>COLOR</th>
<th>% FRG Oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100% 135 mp</td>
<td>0%</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>2</td>
<td>99% 135 mp</td>
<td>1% B</td>
<td>No</td>
<td>0%</td>
</tr>
<tr>
<td>3</td>
<td>98% 135 mp</td>
<td>2% B</td>
<td>No</td>
<td>0%</td>
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<tr>
<td>4</td>
<td>96% 135 mp</td>
<td>1% B</td>
<td>Yes</td>
<td>3%</td>
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<td>5</td>
<td>93% 135 mp</td>
<td>2% B</td>
<td>Yes</td>
<td>5%</td>
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<td>6</td>
<td>100% 135 mp</td>
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<td>0%</td>
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<td>7</td>
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<td>1% C</td>
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<td>8</td>
<td>98% 135 mp</td>
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<td>9</td>
<td>96% 135 mp</td>
<td>1% C</td>
<td>Yes</td>
<td>3%</td>
</tr>
<tr>
<td>10</td>
<td>93% 135 mp</td>
<td>2% C</td>
<td>Yes</td>
<td>5%</td>
</tr>
</tbody>
</table>
## Results w/ Low Melt Point Wax

<table>
<thead>
<tr>
<th>Candle Blend</th>
<th>Burn Rate (g/ hr)</th>
<th>Force (lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>130 mp par + Color</td>
<td>3.60</td>
<td>25.9</td>
</tr>
<tr>
<td>130 mp par + Color + 1% Micro B</td>
<td>3.58</td>
<td>26.7</td>
</tr>
<tr>
<td>130 mp par + Color + 2% Micro B</td>
<td>3.20</td>
<td>30.6</td>
</tr>
<tr>
<td>130 mp par + Color + 1% Micro B + 3% Frag.</td>
<td>3.59</td>
<td>22.9</td>
</tr>
<tr>
<td>130 mp par + Color + 2% Micro B + 5% Frag.</td>
<td>3.72</td>
<td>22.5</td>
</tr>
<tr>
<td>130 mp par + Color + 1% Micro C</td>
<td>2.93</td>
<td>21.2</td>
</tr>
<tr>
<td>130 mp par + Color + 2% Micro C</td>
<td>2.80</td>
<td>24.1</td>
</tr>
<tr>
<td>130 mp par + Color + 1% Micro C + 3% Frag.</td>
<td>3.04</td>
<td>18.5</td>
</tr>
<tr>
<td>130 mp par + Color + 2% Micro C + 5% Frag.</td>
<td>3.07</td>
<td>18.1</td>
</tr>
</tbody>
</table>
## Results with Mid Melt Point Wax

<table>
<thead>
<tr>
<th>Candle Blend</th>
<th>Burn Rate (g/ hr)</th>
<th>Force (lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>135 mp par + Color</td>
<td>3.90</td>
<td>24.9</td>
</tr>
<tr>
<td>135 mp par + Color + 1% Micro B</td>
<td>3.80</td>
<td>29.1</td>
</tr>
<tr>
<td>135 mp par + Color + 2% Micro B</td>
<td>2.78</td>
<td>31.1</td>
</tr>
<tr>
<td>135 mp par + Color + 1% Micro B + 3% Frag.</td>
<td>4.01</td>
<td>24.7</td>
</tr>
<tr>
<td>135 mp par + Color + 2% Micro B + 5% Frag.</td>
<td>4.53</td>
<td>28.0</td>
</tr>
<tr>
<td>135 mp par + Color + 1% Micro C</td>
<td>2.89</td>
<td>28.0</td>
</tr>
<tr>
<td>135 mp par + Color + 2% Micro C</td>
<td>2.82</td>
<td>30.6</td>
</tr>
<tr>
<td>135 mp par + Color + 1% Micro C + 3% Frag.</td>
<td>3.06</td>
<td>23.5</td>
</tr>
<tr>
<td>135 mp par + Color + 2% Micro C + 5% Frag.</td>
<td>3.13</td>
<td>24.0</td>
</tr>
</tbody>
</table>
Recommendations

- Must be balanced with major candle components and additives
- Micro wax for modifying opacity
  - Varying micro % when balancing the candle formula will alter color values slightly
  - Use of 3-5% will improve fragrance retention and opacity
- Micro wax for molded candle applications
  - 1% hardening grade to improve structural/heat integrity
  - 1% to improve fragrance retention, more if used also for heat resistance
- Micro wax for compression applications
  - Compression applications: 1-2% will improve candle strength as well as fragrance retention
  - Wax used must balance needle pen value which is hard enough to improve strength but not too hard to adversely affect compressibility
- Micro wax for Jar Candles
  - 5% laminating grade will improve adherence and allow improved fragrance retention
## Selecting Microcrystalline Wax vs. Other Candle Additives

<table>
<thead>
<tr>
<th>Compare To</th>
<th>Cost/lb</th>
<th>Processing Ease</th>
<th>Molecular Weight</th>
<th>% Required</th>
<th>Compat. w/ paraffin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Veg Wax</td>
<td>↓</td>
<td>↓</td>
<td>-</td>
<td>-</td>
<td>↓</td>
</tr>
<tr>
<td>High MP Fischer Tropsch</td>
<td>↑</td>
<td>↓</td>
<td>↑</td>
<td>↓</td>
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</tr>
<tr>
<td>Modified Polyethylene</td>
<td>↑</td>
<td>↓</td>
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