SILICONE DISPERSANTS FOR FRAGRANCES IN CANDLES

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GE ADVANCED MATERIALS

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CANDLES & FRAGRANCE

Candle industry research indicates the most important factors affecting candle sales are:

scent    color    cost    shape

¾ of people who purchase candles say that fragrance is the most important characteristic.*

Desire for increased hot/cold throw has led to increased fragrance loadings which will:

• increase syneresis
• increase mottle
• lead to problems with combustion

*National Candle Assn. Web Site
ORGANOMODIFIED SILICONES

• Widely used in skin and hair care as aesthetic ingredients and as dispersants, emulsifiers, and foam modifiers

• In agricultural applications as an adjuvant for wetting and spreading of active ingredients

• In coatings as flow & leveling agents, and for slip, air release, and pigment dispersibility

• For home care as wetting agents in auto dish, fabric care, and now.......for fragrance dispersibility in CANDLES
SILICONE SURFACTANTS
(not be be confused with polydimethylsiloxane)

Broad class of polyalkyleneoxide modified polydimethylsiloxane:

3 Structural Types:

- **Pendant**
- **Trisiloxane**
- **Linear**

This flexibility results in tremendous structural diversity!
# METHOD DEVELOPMENT
(Design a More Efficient Candle)

**HYPOTHESIS:** Increased fragrance dispersibility will improve the appearance, reduce syneresis, increase throw, & allow for increased fragrance loadings or get the same throw from a lower fragrance loading.

**TECHNICAL APPROACH:** Use organo-modified silicones for better compatibility of fragrance in candle matrix.

**MEASURABLES:**
- change in color ($\Delta E$) to measure appearance of candle
- use fl. ht., burn pool, ROC to measure impact of Si on combustion
- temp. cycling to quantify syneresis
- GC/MS to measure fragrance throw; confirm with panel assessment

**MODEL FORMULATION:**
Exxon Mobil 140 paraffin wax
Cotton core AP44-20-18C wicks
Fragrance:
- various
- 10-15 wt% concentration
Organomodified silicone:
- various
- 0.5 wt %
- mixed with fragrance
PROOF OF CONCEPT-VANILLA

VANILLA
• Extremely popular
• Is problematic
• Vanilla is simple compositionally

INITIAL VALIDATION WORK
Screened a series of structures to determine if they would
• disperse vanilla
• affect flame height
• burn rate
• sooting behavior

15wt% vanilla
0.5wt% Formasil* 500
84.5wt% paraffin wax

* Formasil is a registered trademark of the General Electric Company
PROOF OF CONCEPT - VANILLA

- Sample A has not affected the flame or soot relative to the control with or without fragrance
- It does not improve dispersibility

- Dispersants B & C have reduced the flame height or large melt pools
- D (Formasil* 500) has an ex. appearance; very close to control without fragrance

- F provides excellent dispersibility but has impacted flame height
DEMONSTRATION OF DISPERSIBILITY

- Candles made with and without Formasil* 500
- Cut candles into top/middle/bottom sections
- Remelted sections
DISBERSIBILITY QUANTIFIED

In our experimental design the color is due only to the fragrance and therefore distribution of color relates to distribution of fragrance.

IMPROVEMENT IN CHANGE IN COLOR

Measure ΔE across bottom, middle, & top sections of candles made with 15% vanilla & find that color is more uniform when Formasil 500 is used.

HunterLab
Color Measurements (L*A*B)
DISPEROSIBILITY QUANTIFIED

ADDITIVE DISTRIBUTION
Confirmed that the additive was uniformly distributed in a similar experiment where candle sections were digested & assayed for %Si

Additive Distribution

% Si

Top | Middle | Bottom
--- | --- | ---

0.45 | 0.45 | 0.45

Dispersibility quantified.
SYNERESIS (BLEED)

HYPOTHESIS: if fragrance is more uniformly distributed throughout matrix syneresis will be reduced

WE QUANTIFY SYNERESIS BY:
• wrapping candles in preweighed absorbent tissue ($T_1$)
• subjecting them to accelerated aging by temperature cycling over a 24 hr. period
• reweighing tissue ($T_2$)
SYNERESIS TEST SHOWS IMPROVED FRAGRANCE RETENTION

In candles containing 15% vanilla with & without 0.5% Formasil* 500 we see a dramatic reduction in vanilla fragrance loss.

![Fragrance Loss Graph]

In candles containing 10% Blood Valencia with & without 0.5% Formasil 520 we see a 45% reduction in citrus fragrance loss.

![Fragrance Loss Graph]
IMPACT OF SILICONE DISPERSANT ON FLAME HEIGHT

Height of the flame from the base of the flame at the wax pool to the highest visible point of the flame and reported in cm.*

*Atkins & Pearce Test Method
IMPACT OF SILICONE DISPERSANT ON BURN POOL
Average of 2 readings measured at the same time as flame height and reported in cm.*

*Atkins & Pearce Test Protocol
NO DETECTABLE IMPACT OF FORMASIL* 520 ON COMBUSTION

- Melt Pool 4hr
- Melt Pool 3hr
- Melt Pool 2hr
- Melt Pool 1hr
- Flame Height 4hr
- Flame Height 3hr
- Flame Height 2hr
- Flame Height 1hr

Legend:
- Wax control
- 10% Blood Valencia
- 10% Bl. Val w/ Formasil* 520
NO DETECTABLE IMPACT OF FORMASIL* 500 ON COMBUSTION

<table>
<thead>
<tr>
<th></th>
<th>Wax Control</th>
<th>6% Poundcake</th>
<th>6% Poundcake/Formasil* 500</th>
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<tbody>
<tr>
<td>Melt Pool 4 hr</td>
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<tr>
<td>Melt Pool 3 hr</td>
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<tr>
<td>Melt Pool 2 hr</td>
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<tr>
<td>Melt Pool 1 hr</td>
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<tr>
<td>Flame Height 4 hr</td>
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<td>Flame Height 1 hr</td>
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</tbody>
</table>

cm.
SILICONE DISPERSANTS HAVE LITTLE IMPACT ON RATE OF COMBUSTION (ROC)

**ROC** is the amount of wax consumed over a fixed period of time.

**Method:**
- Weigh candle
- Burn candle
- Reweigh remaining mass
- Divide difference in mass by the exact burning time
- Report in gm/hr.*

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### Rate of Combustion

<table>
<thead>
<tr>
<th>Compound</th>
<th>GM/HR</th>
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<tbody>
<tr>
<td>6% Poundcake/Formasil* 500</td>
<td></td>
</tr>
<tr>
<td>6% Poundcake</td>
<td></td>
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<tr>
<td>Exxon Mobil 140 Wax</td>
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</tbody>
</table>

*Atkins & Pearce Test Protocol*
BETTER FRAGRANCE DISPERSION IMPROVES THROW

HYPOTHESIS: fragrance that is more uniformly dispersed throughout a matrix will provide improved throw as opposed to incompatible fragrance that is sitting in pools or pockets at candle bottom.

ASSESSMENT: qualitative panel assessments support that there is stronger scent from candles made with organomodified silicone than corresponding controls without. This is an inherent property of the candle formulation. Confirm by GC/MS head space analysis.
HEAD SPACE ANALYSIS METHOD

• Remove 0.5 gm of candle using #2 cork borer
• Place in 20 ml head space vial and seal
• Samples were equilibrated at 35°C and 140°C and analyzed with SPME (Solid Phase Microextraction)
  – (5 min. SPME exposure in headspace followed by GC analysis)

Sampling of Products

SPME Needle

Sample

SPME Needles coated with PDMS/Carboxen to absorb volatiles from atmosphere.
HEAD SPACE ANALYSIS CONFIRMS ENHANCED THROW

• Collected GC head space analysis on key chemical components of the fragrance
• Candles containing Formasil* 520 and 10% Bl Valencia have improved hot throw
HEAD SPACE ANALYSIS CONFIRMS ENHANCED THROW

• Collected GC head space analysis on key chemical components of the fragrance
• Candles containing Formasil* 500 and 10% Carmel Apple have improved hot & cold throw
FORMASIL DISPERSANTS CAN IMPROVE FRAGRANCE THROW

• GC headspace analysis shows enhanced hot throw for Formasil* 520

• GC headspace analysis is inconclusive for cold throw
  – 9/10 sensory panelists selected candle containing Formasil dispersant as having better cold throw

• GC headspace analysis shows enhanced hot & cold throw for Formasil 500

FORMASIL SILICONE DISPERSANTS ALLOWS CANDLE MANUFACTURES TO USE LESS FRAGRANCE FOR THE SAME ODOR!
SIMILAR FRAGRANCES EFFECTIVELY DISPERSED BY THE SAME SILICONE

HYPOTHESIS: Fragrances that are similar compositionally can be effectively dispersed by the same organomodified silicone.

PROOF OF CONCEPT: Berry fragrance from 2 different suppliers

Similarities:
• Compositional overlap of major components (14% vs. 25%)
• Limonene is the major ingredient in frag #1; 2nd most abundant in frag #2

Differences:
• Carrier: 74% diisonyl phthalate vs 35% IMP

Rate of Combustion
SYNERESIS TEST AGAIN SHOWS IMPROVED FRAGRANCE RETENTION

![Graph showing fragrance retention comparison between control and Frag/Formasil 520 for Tropical Berry and Berry I. The graph indicates improved fragrance retention for Frag/Formasil 520 compared to the control.](image-url)
FORMASIL DISPERGANTS IMPROVE FRAGRANCE THROW

6% INCREASE HOT THROW

12% INCREASE HOT THROW
CONCLUSIONS

• Use of organomodified silicones improves fragrance dispersion

• Candles prepared with Formasil* silicone dispersants showed:
  – enhanced throw with a variety of formulated fragrances
  – reduced syneresis
  – reduced mottle
  – little or no impact on combustion

• Formasil Organomodified Silicones – An Alternative to Higher Fragrance Loadings for Improved Fragrance Performance

GE Has Filed a Patent on This Technology
ACKNOWLEDGEMENTS:

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