Syneresis (Bleed) in Candles
A Petroleum Wax Perspective

THE INTERNATIONAL GROUP, INC.

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By: THE INTERNATIONAL GROUP, INC.—Candle Development Team September 2001

ABSTRACT: *The introduction of mottled or highly fragranced candles has lead to an increase in syneresis or bleed in petroleum wax candles. The paper will cover the utilization of additives and process control to impact syneresis, and an overview of the test procedures developed for this study.*

The term syneresis, more commonly referred to as bleed, in candles occurs when a liquid component separates from the solid wax mass (Figure 1). With the candle industry's move towards higher fragrance loading and with many candle producers trying to achieve a "mottled" look in candles, the incidents of bleed have become much more prevalent. The separation of liquid (i.e. fragrance oil, liquid dye, soft wax components or mineral oil) can have several points of origin. Bleed is often caused by non-optimized candle production or an incompatibility of additive(s), fragrance or solvent with the base wax. A candle's thermal life cycle may also create conditions conducive to bleed.

Bleed results in candles developing a greasy surface, and in extreme cases, droplets appearing on the outer surface of the candle. Candle bleed negatively impacts the look and performance of candles. Bleed can cause undesired liquid transfer onto the consumer's fingers or candle package, or leave handling marks on the candle surface. In a container candle, liquid migration can leave "pockets" between the wax and the container walls. Bleed has also been linked to the occurrence of undesired surface crystallization or mottling as the liquid phase migrates to the candle's exterior. This phenomenon can also result in compromised fragrance cold and/or hot throw in the finished product as fragrance migrates to the candle surface.

The study of bleed presents some formidable challenges to producers and suppliers alike. Very few test procedures exist which allows consistent quantification of bleed. Secondly, candle bleed may occur immediately or manifest itself over several weeks or months.

The goal of IGI's Candle Development Team's research was to explore the mechanisms of bleed and to understand how various additives, fragrance types, and processing / handling parameters affect the degree to which a candle bleeds. To facilitate this work the team developed unique standardized test procedures and established baseline data to serve in on-going development work in creating enhanced candle systems.

Development of Tests for Measuring Candle Bleed

To assess comparative levels of bleed, and the impact of various additives, IGI's Candle Development Team required the development and implementation of two test procedures.

The first test procedure enabled the definitive quantification of bleed between various candle formulations. This critical technique would permit researchers to gather reproducible bleed data produced by variations in the components and processing of candles.

The second test method addressed the problem with candle bleed occurring at varied stages of a candle's life cycle. It was essential to measure the tendency of a candle system to bleed over its entire lifecycle. This test proved extremely valuable in highlighting the need for post-production monitoring of all candle products.

Quantification of Bleeding

IGI evaluated two techniques designed to quantify bleed in candles; one designed to measure weight update by a substrate, the other weight loss by the candle itself.

Figure 1: *Evidence of Syneresis in a Container Candle*

Weight uptake by a substrate

Candles were wrapped in an absorbent substrate and then placed in sealed polyethylene bags. After a predetermined time and conditioning the increase in weight of the substrate was measured. Weight gain was considered to be a result of migrating liquid.

Weight loss by candles

Candles were weighed directly after cooling. The candles were then wiped dry and reweighed after a specified time. Weight gain is considered to be a result of migrating liquid.

Good correlation for both methods was found for most candles (see Figure 2). Citrus fragranced candles were the exception to the rule as they exhibited brittleness and measuring weight loss proved difficult due to product crumbling and loss (Figure 3). For this reason IGI has chosen to utilize substrate weight gain in the measurement of candle bleed in all systems.

Accelerated Aging

The second test developed for this investigation was designed to accelerate bleeding through temperature cycling. This technique would assess the tendency of a candle to bleed over an extended period of time.

The accelerated aging procedure used on the candles is outlined below.

a. Air-cooling for 2 hrs after pouring
b. Weighing
c. Storage at 4°C (39°F) for 1 hr
d. Storage at 40°C (104°F) for 1 hr
e. Storage at 4°C (39°F) for 1 hr
f. Storage at 22°C (72°F) for 19 hrs
g. Weighing 24 hrs after original pour

Figure 4 illustrates substrate weight gain by fragrance type.

Figure 2: Low mp wax: Substrate weight gain vs votive weight loss

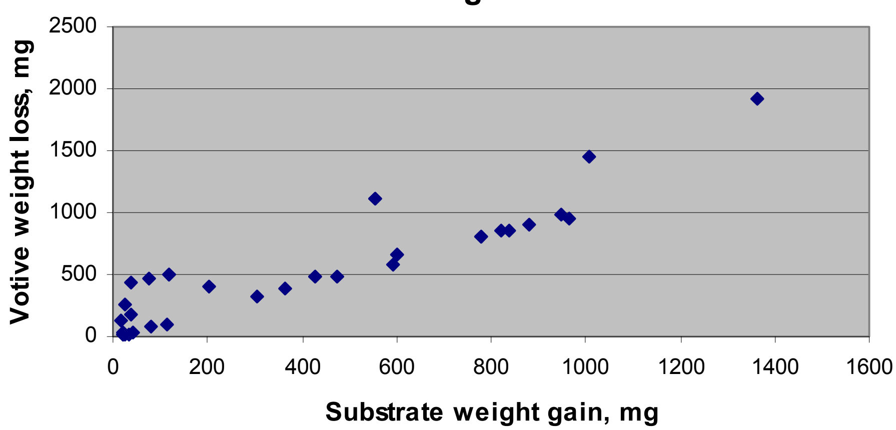


Figure 4: Mid mp wax: Bleeding with fragrances at 20% level

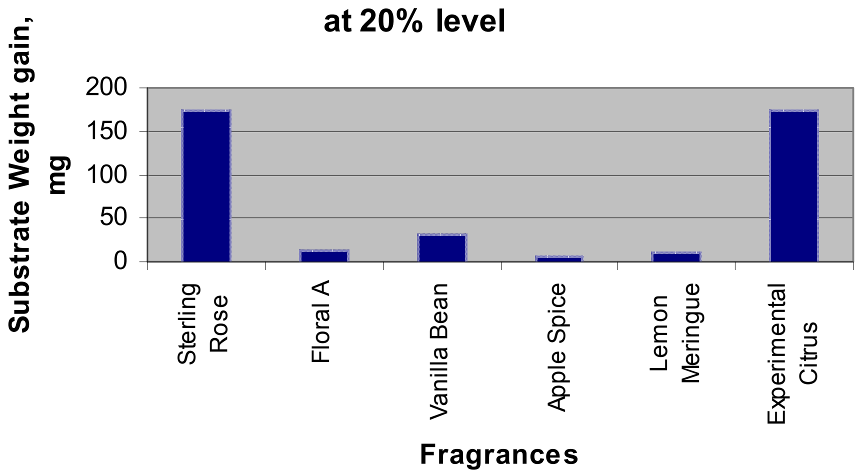


***Figure 3**: Cross-section of a votive candle showing brittleness*

Factors Affecting Bleed

Bleed and Wax Type

IGI utilized straight cut paraffin waxes as the base wax for each evaluation. While the findings regarding fragrance and additive type apply primarily to the system investigated, the findings should be considered of importance to the industry. Experimental conditions were standardized to reduce variation in the experiments and allow for more effective comparisons:

Candle type: Votive
Wax types:

- Straight cut high-melt point (163°F) paraffin
- Straight cut mid-melt point (145°F) paraffin
- Straight cut low-melt point (126°F) paraffin

Fragrance types (added at 20% level to exaggerate bleed):

- Floral
- Spice
- Citrus

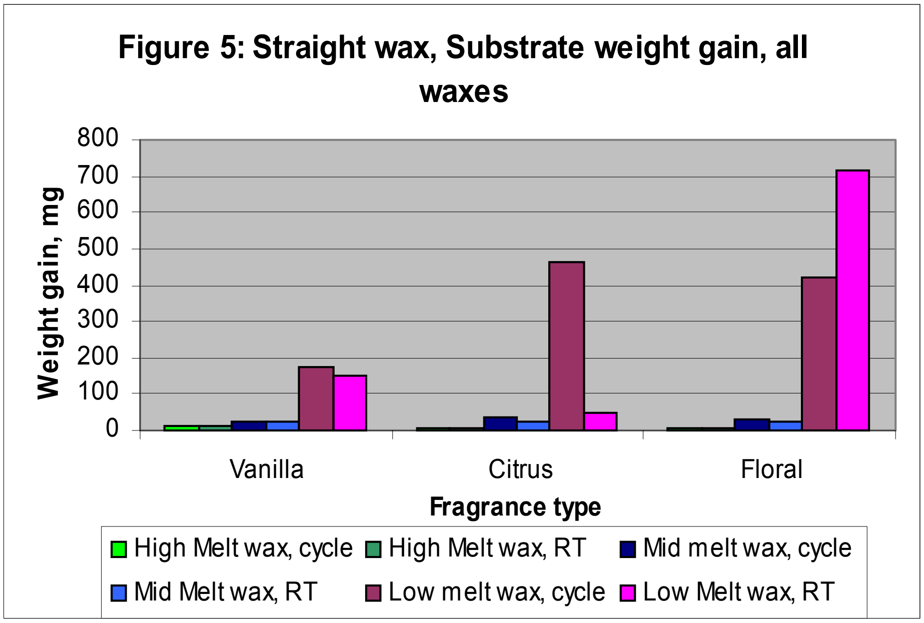
Dye type: Dry Powder—Yellow
Processing Conditions:

- 180°F Pour Temperature
- 72°F Cooling Temperature

After the baseline evaluations were completed, a selection of wax additives were utilized in conjunction with each fragrance and wax type to assess their effect on bleeding.

For each candle system the level of candle bleed was determined via the substrate weight gain method as well as by candle weight loss. Candle shrinkage was also measured to determine if there was a significant correlation between shrinkage and bleed.

Results Observed:
Results from this experiment indicate that for the straight cut paraffin waxes used, higher melting point waxes exhibit less bleed (Figure 5).



Impact of Candle Additives

The impact on bleed of certain candle additives (Table 1) was explored.

The low and mid melt point paraffin waxes were screened using all combinations of fragrance and additives. The initial evaluation using high melt point paraffin wax displayed minimal bleed, so it was not included in the additive screening.

***Table 1**: Additives Screened and Addition Level Used*

Additive	Level Used
Microcrystalline wax (IGI Microsere 5702)	2%
Stearic Acid (Distilled 90)	5%
Polyethylene Wax	1%
Buty Stearate	5%
Isostearyl Alcohol	5%
Castor wax	10%
Perlatum 310 (IGI)	10%
Sodium stearate	3%
Ethylene Vinyl Acetate	1%
Vybar 103 (Baker Petrolite)	1%
Paraflint H1 (Moore and Munger)	1%

Summary of Findings*

The following observations were made and conclusions drawn in relation to bleed:

- Additives had different effects for each of the fragrances.
- Additives had greater effect on low melt point systems than mid melt point systems.
- Temperature cycling had the greatest effect on the low melt wax with citrus fragrance.
- No correlation was found between shrinkage and bleeding.

*The findings in this study were limited to straight cut paraffin waxes rather than an optimized wax blend. Additional studies indicate that a wax blend exhibits less bleed than was observed with straight cut high melt paraffin waxes.

System Optimization

Using the base experimental data derived from both the fragrance and additive portions of the study, The IGI Candle Development Team worked to minimize bleed in candle systems. The fragrance/additive systems used for this study were as follows:
1. Spice Fragrance (Vanilla Bean) and Microsere 5702 (IGI) additive
2. Floral Fragrance (Sterling Rose) and Polyethylene AC-6 (Allied Signal) additive

A factorial experimental design (L9 Taguchi Array, Table 2) was used for the optimization. This allows the testing of four production parameters at three different levels. The array design method maximizes information while minimizing blending requirements. In this work wax pour temperature, additive addition rate, wax melt point and candle cooling rate were tested at low, medium and high levels (see Tables 3 & 4) to determine the impact on bleed for the finished product.

The testing levels for the spice (vanilla) fragrance/additive (microcrystalline wax) system are found in Table 3.

Summary of Findings

The response chart for the cycled Vanilla/microcrystalline wax system (Figure 6) indicates that all the factors investigated have equivalent effect in terms of magnitude. In summary:

- Addition of microcrystalline wax can be optimized.
- Addition levels of microcrystalline

Table 2: *L9 Taguchi Array*

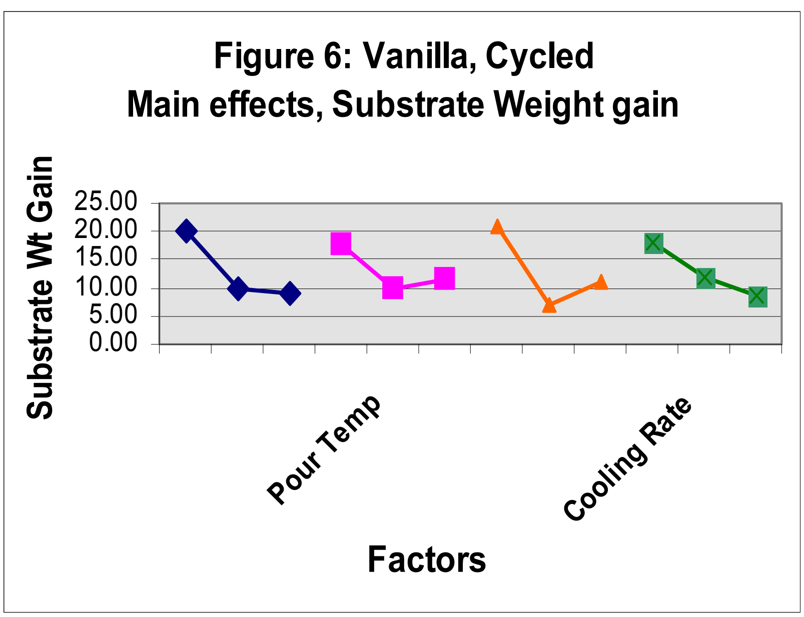
Run	Add^n level	Pour Temp	Wax mp	Cooling rate
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Table 3*: Vanilla /Micro System - Factors Optimized and Levels Tested*

Factors	Level	Level	Level
	Low (1)	Medium (2)	High (3)
1. Micro level	2%	5%	8%
2. Pour temp	170°F	180°F	190°F
3. Wax type	Low mp, 126°F	Mid mp, 145°F	High mp, 163°F
4. Cooling Rate	39°F	72°F	104°F

Table 4*: Floral /PE System—Factors Optimized and Levels Tested*

Factors	Level	Level	Level
	Low (1)	Medium (2)	High (3)
1. PE Add. level	0.50%	2%	4%
2. Pour temp	170°F	180°F	190°F
3. Wax type	Low mp, 126°F	Mid mp, 145°F	High mp, 163°F
4. Cooling Rate	39°F	72°F	104°F



- wax and reduction in bleed is not a linear response (from 5% to 8% produces minimal change).
- Wax pour temperature of 180°F is optimal.
- Mid-melt pt wax yields a satisfactory candle.
- The slowest cooling rate minimizes bleed.

The testing levels for the floral (Sterling Rose) fragrance/additive (polyethylene wax) system are found in Table 4. The corresponding response chart is found in Figure 7.

Summary of Findings

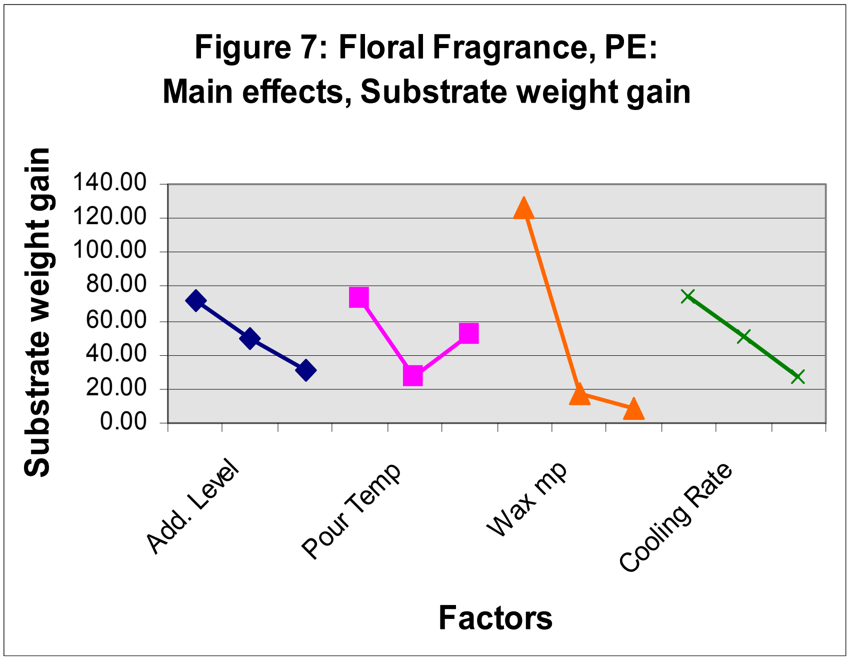
Unlike the previous system, factors investigated in the floral/polyethylene candles did not exhibit equivalent effects in terms of magnitude. The critical factor for making a successful candle with this floral fragrance was the choice of the base paraffin wax. In summary:

- High and mid melt point waxes bled much less than the low melt point wax.
- Response to the additive level was linear (↑ additive results in ↓ bleed).
- Wax pour temperature of 180°F is optimal.
- The slowest cooling rate minimizes bleed.

In conclusion, the optimum conditions for the two systems studied, though similar, were not identical.

Mottled Candles

With mottled candles becoming more and more prevalent we must consider how the results from this investigation can be applied to bleed reduction in this candle type. Additives found to reduce bleed in this study have been proven to reduce the amount and consistency of mottling. (Ref: ***Paraffin Mottling in Candlemaking***, September 1999, paper by IGI) Optimizing production conditions (pour and cooling temperatures) may reduce the bleed in mottled candles. The most important factor to optimize in all candle systems appears to be the compatibility of the fragrance with the base wax.



Conclusions

Syneresis or bleed in candles results from a separation of a liquid phase from the solid wax mass. This phenomenon is of critical importance because it can detract from a candle's appeal by reducing its visual appeal, handling characteristics, and fragrance throw. The following factors were observed and conclusions drawn in relation to bleed:

- Using a higher melt point base wax (straight cut paraffin wax) reduces the tendency of a candle to bleed.
- Variation in storage and transport temperatures may cause or aggravate candle bleed.
- Additives (microcrystalline wax, polyethylene, petrolatum, Vybar, and EVA) can effectively reduce candle bleed.
- Additives function differently depending on fragrance and wax selection.
- Production conditions must be optimized for each wax-fragrance system.

IGI Candle Industry Commitment

IGI and its Candle Development Team is committed to understanding the general science, chemistry and physics involved in candle making and burning. We are dedicated to applying practical knowledge and scientific principles to wax formulating so to provide technically sound and innovative candle solutions to our customers.

Special thanks to the IGI Candle Development Team members responsible for the work behind this paper including:
Ian Davie, Juliana Dragomir, Sean McColl, Baz Mistry and Joey Viljoen

THE INTERNATIONAL GROUP, INC.

About IGI

THE INTERNATIONAL GROUP, INC. (IGI) is a customer driven corporation committed to understanding and satisfying customer's needs.

Founded in 1945, IGI has grown from a small independent Canadian refinery to a global leader in the refining and compounding of petroleum waxes, amorphous (atactic) polypropylene (APP) products and specialty ployolefin compounds.

Today, IGI owns and operates the largest independent, ISO 9001 registered, petroleum wax refinery/blending plant in North America. This is in addition to terminal and blending facilities, warehouses and sales offices located throughout North America.

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