



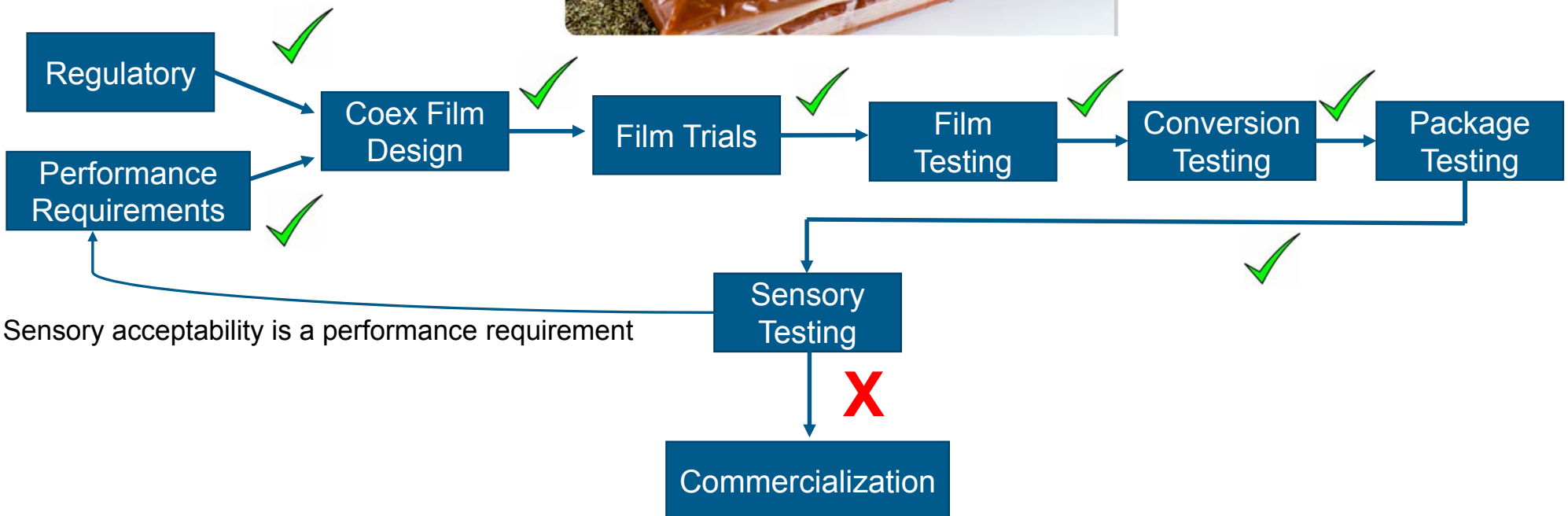
Sensory Evaluation of Polyethylene Sealants and Additives used in Flexible Food Packaging

Dan Ward
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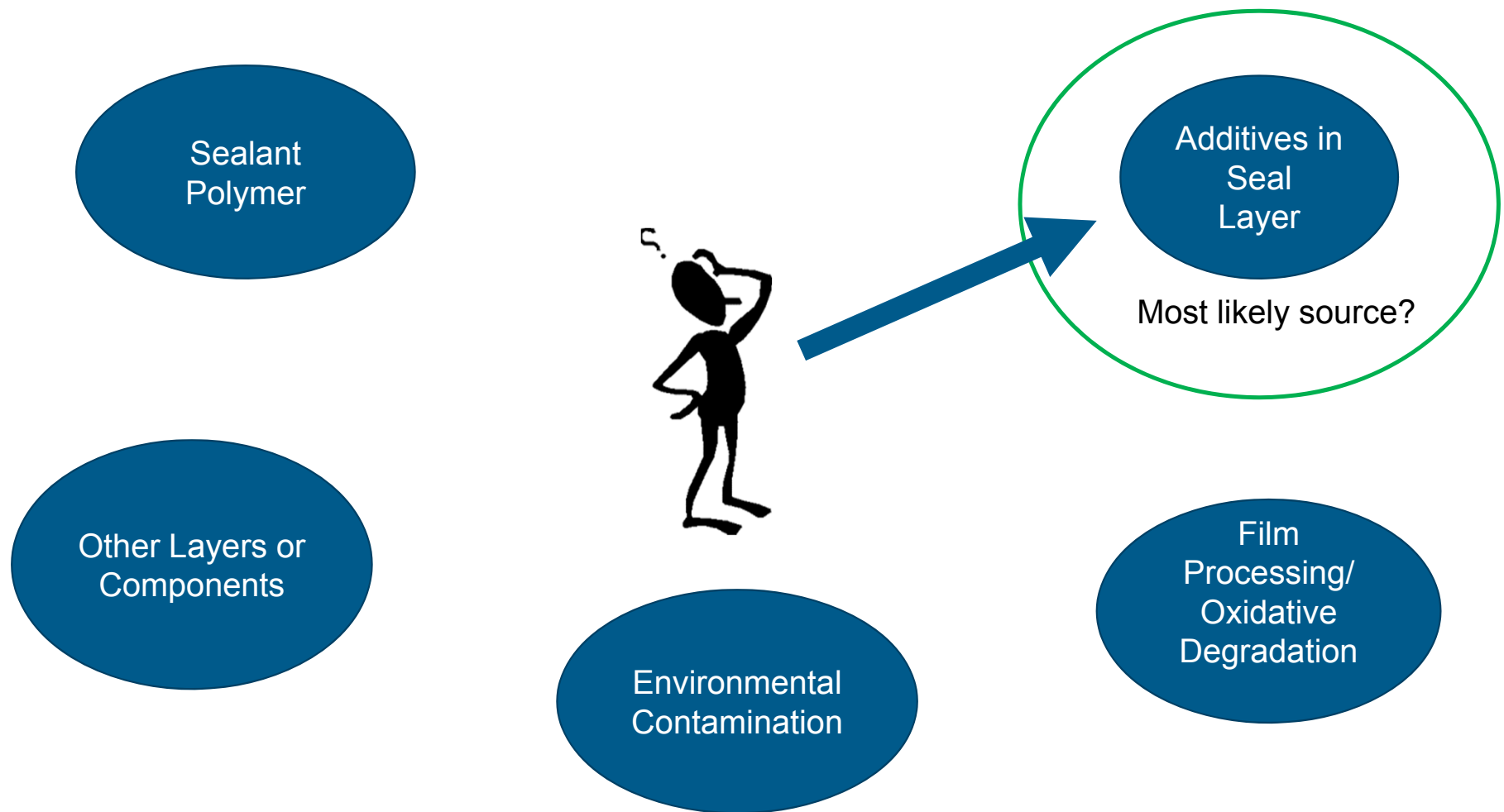
April 16, 2024

Food Packaging Development Stages

Application Large Liquid Food Pouch with High Performance Sealant



Possible Root Causes of Sensory Failure

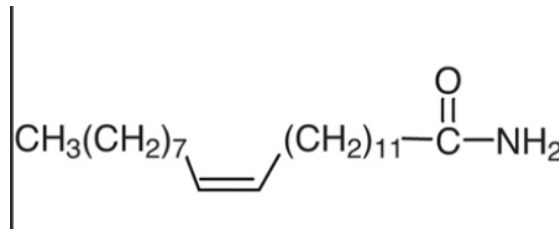


Slip and Antiblock Masterbatches

*Essential Additives for most Seal-able Films
Functions, not distinct chemicals*

Primary Function of Slip: Reduce Surface Coefficient of Friction

- General Chemistry of Functional Component – Fatty acid amide
- Other Masterbatch Ingredients: Carrier resin, stabilizers, dispersing agents, etc...



Primary Function of Antiblock Masterbatches: Reduce Film to Film Adhesion

- General Chemistry of Functional Component – Inorganic Minerals





Study 1 - Sensory Effects of Slip and Antiblock Masterbatches

OBJECTIVE: Measure and compare the effects of two distinct slip and antiblock masterbatches on the sensory characteristics of water in monolayer film pouches.

TEST FILMS and MATERIALS

- 3.5 mil monolayer blown films
- 3 Base Resins:
 - LDPE, Octene LLD Control, and EVA (9% VA)
- 2 Slip and 2 Antiblock masterbatches: “Standard” (Control) and “Low Odor” (Test)
 - Slip Masterbatches blended at 5% for 3000 ppm Slip.
 - Antiblock Masterbatches blended at 1% for 5000 ppm AB



SENSORY TESTING - METHOD DEVELOPMENT

PANEL LEADER PRE-SCREENING USING ASTM E1870-11: Odor and Taste Transfer from Polymeric Packaging Films

- Several samples were highly objectionable and overwhelming.
 - *Film Surface to Water Volume Ratio was reduced.*
- Filtered water was not a suitable control.
 - *Switched to Poland Spring Bottled Water*
- Panelist fatigue when more than 6 samples per set were evaluated
- Specific Sensory Attributes could be differentiated.



Test Method Modifications

- Pouch Dimensions, Water, number of samples per test set...
- Re-evaluations after modifications gave better differentiation and an acceptable range of scores

Sample Scoring and Sensory Attributes

Degree of Difference from the Known (Blank Water) Control Sample.

LESS THAN CONTROL							Like Control	MORE THAN CONTROL						
Very Much Less	Much Less	Moderately to Much Less	Moderately Less	Slightly to Moderately Less	Slightly Less	Very Slightly Less	No Difference	Very Slightly More	Slightly More	Slightly to Moderately More	Moderately More	Moderately to Much More	Much More	Very Much More
-7	-6	-5	-4	-3	-2	-1	0	1	2	3	4	5	6	7

Sensory Attributes

Attribute Intensity										
None		Slight		Moderate			Strong			Very Strong
0	1	2	3	4	5	6	7	8	9	10

- **Taste**
 - Bitter – taste on the tongue associated with caffeine in solution.
 - Sour – taste on the tongue associated with acids such as citric acid in solution.
- **Mouth Feel**
 - Oily mouthfeel/thickness – the oily or waxy mouthfeel coating feeling on the oral surfaces
 - Astringent – the drying and puckering sensation on the oral surfaces similar to strong black tea (tannins)
 - Irritating – the painful sensation related to prickly, stinging, or tingling sensation on the tongue
- **Aromatics**
 - Crayon – aroma associated with unscented candle wax and crayons
 - Soapy – aroma associated with unscented liquid soaps
 - Heated plastic – aroma associated with old water bottles subjected to heat and plastic shower curtains
 - Chlorine – aroma associated with highly chlorinated water such as swimming pool water
 - Earthy/Musty – aroma associated with earth such as potting soil.

“Flavor Profile” = combined taste, mouthfeel and aromatics

Pouch Sample Preparation and Sensory Test Protocol

- Films sealed and converted into pouches within 24 hours of production
- Pouches wrapped in foil liners and shipped to Sensation Research.
- Pouches filled with 1.5 liters Poland Spring Water and resealed with minimal headspace
- Filled pouches stored at 72° F for 28 days
- 90 milliliters of test water poured into brandy snifters and covered with convex glass.



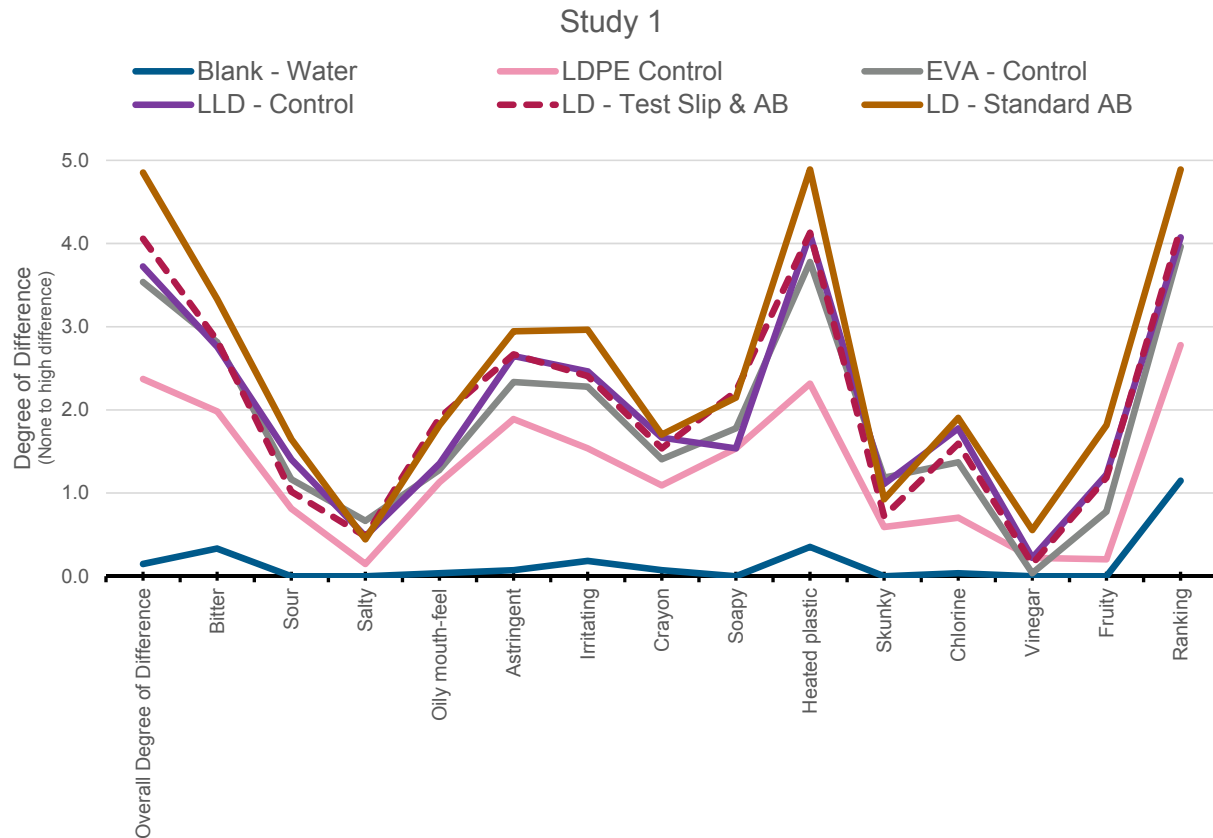
Difference from Water Control for Sample Set 1

Set 1 Additive Study; N = 9 x 3 Evaluations per Sample	Blank – Water Blind Control	LDPE Control	EVA - Control	LLD - Control	LD - Test Slip & AB	LD - Standard AB	HSD at 95%	Prob
Overall Degree of Difference	0.1 ^c	2.4 ^b	3.5 ^{ab}	3.7 ^{ab}	4.1 ^a	4.9 ^a	1.37	0.0001
Bitter	0.3 ^c	2.0 ^b	2.8 ^{ab}	2.8 ^{ab}	2.8 ^{ab}	3.3 ^a	1.16	0.0001
Sweet	0.3	-0.3	-0.3	0.2	0.6	0.5	1.09	0.0684
Sour	0.0 ^b	0.8 ^{ab}	1.2 ^a	1.4 ^a	1.0 ^a	1.6 ^a	0.87	0.0001
Salty	0.0 ^b	0.1 ^{ab}	0.7 ^a	0.5 ^{ab}	0.5 ^{ab}	0.4 ^{ab}	0.55	0.0081
Oily mouth-feel	0.0 ^b	1.1 ^{ab}	1.3 ^a	1.4 ^a	1.9 ^a	1.8 ^a	1.12	0.0002
Astringent	0.1 ^b	1.9 ^a	2.3 ^a	2.6 ^a	2.7 ^a	2.9 ^a	1.12	0.0001
Irritating	0.2 ^c	1.5 ^b	2.3 ^{ab}	2.5 ^{ab}	2.4 ^{ab}	3.0 ^a	1.12	0.0001
Crayon	0.1 ^b	1.1 ^{ab}	1.4 ^a	1.7 ^a	1.5 ^a	1.7 ^a	1.14	0.0008
Soapy	0.0 ^b	1.5 ^a	1.8 ^a	1.5 ^a	2.2 ^a	2.1 ^a	1.16	0.0001
Heated plastic	0.4 ^c	2.3 ^b	3.8 ^a	4.1 ^a	4.1 ^a	4.9 ^a	1.39	0.0001
Skunky	0.0 ^b	0.6 ^{ab}	1.2 ^a	1.1 ^{ab}	0.7 ^{ab}	0.9 ^{ab}	1.12	0.0345
Chlorine	0.0 ^c	0.7 ^{bc}	1.4 ^{ab}	1.8 ^a	1.6 ^{ab}	1.9 ^a	1.02	0.0001
Vinegar	0.0 ^b	0.2 ^{ab}	0.0 ^b	0.2 ^{ab}	0.1 ^{ab}	0.6 ^a	0.49	0.0255
Fruity	0.0 ^c	0.2 ^{bc}	0.8 ^{abc}	1.2 ^{ab}	1.2 ^{ab}	1.8 ^a	1.06	0.0001
Ranking (Friedman's)	1.1 ^c	2.8 ^b	4.0 ^{ab}	4.1 ^{ab}	4.2 ^{ab}	4.9 ^a	NA	0.0001

Within an attribute, products sharing a common letter are not significantly different at the 95% confidence level as tested by ANOVA with Tukey's HSD for Post Hoc comparisons.

Lowest in attribute	Not Sig. Different from Lowest	Not Sig. Different from Highest or Lowest	Not Sig. Different from Highest	Highest in attribute
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Relative Mean Differences for Study 1



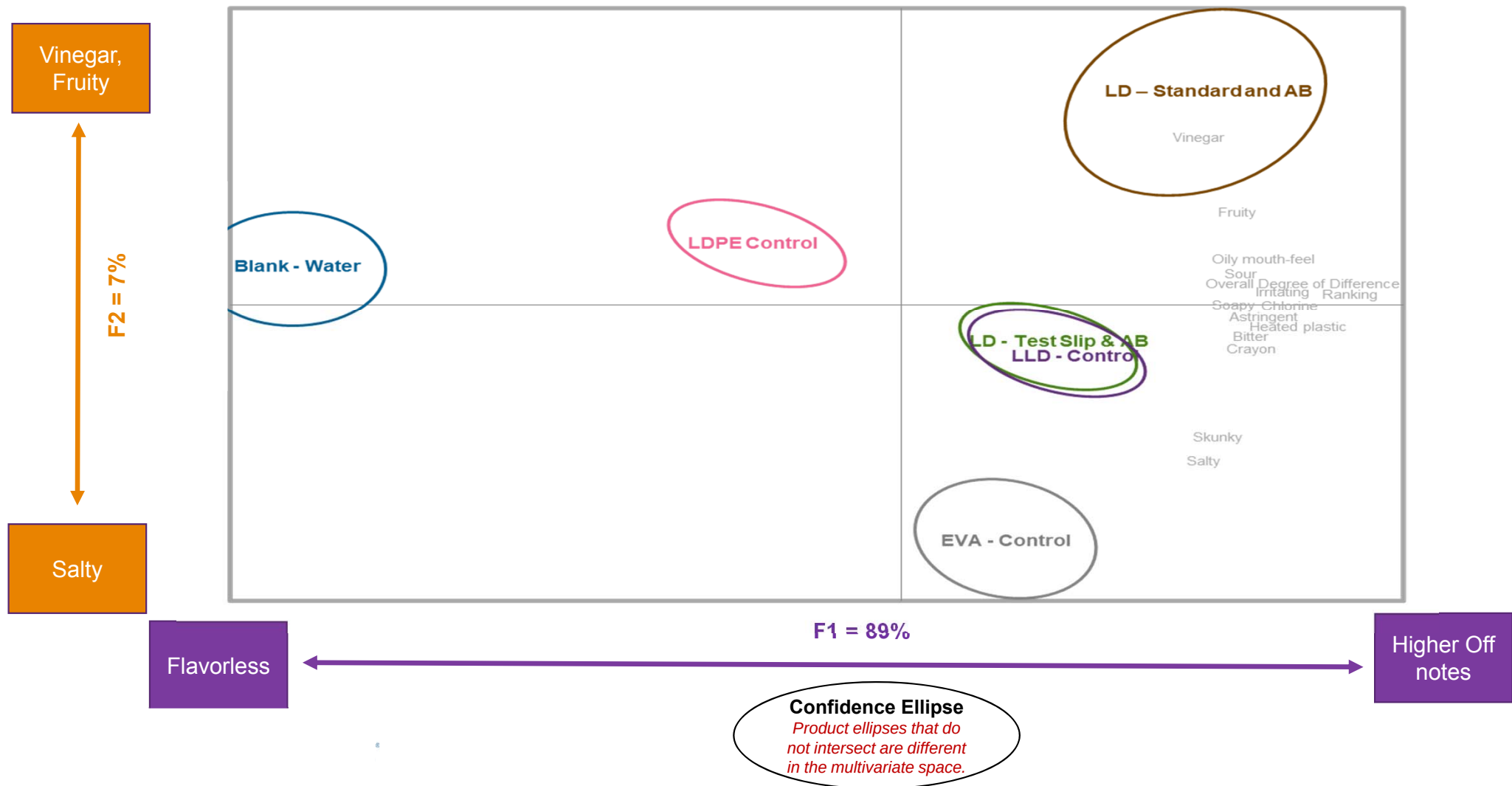
The **Blank-Water** is nearly zero for all attributes and has the lowest rank (1=least off-notes).

In general, the following trends are noticed of at least 0.5 points (or more) difference between the next closest sample:

The **LDPE Control** is closer to the **Blank-Water** than the other samples for overall degree of difference, bitter, astringent, irritating, heated plastic, chlorine, fruity and ranking.

The **LD - Standard AB** is most different in overall degree of difference, bitter, irritating, heated plastic, fruity and ranking.

PCA and DoD Map for Additive Study - Set 1



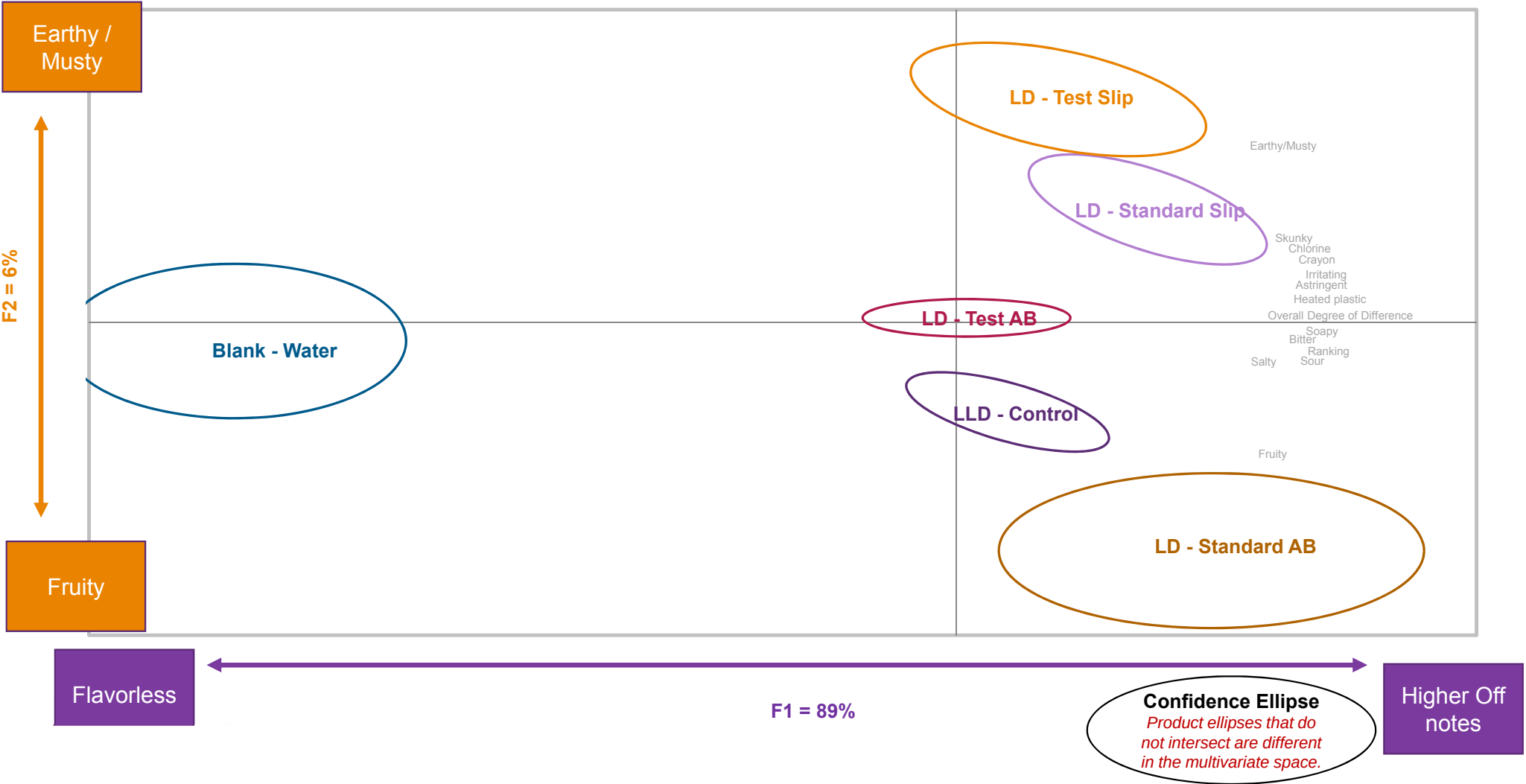
Difference from Water Control for Sample Set 2

Set 2 Additive Study N = 9 x 3 Evaluations per Sample	Blank - Water	LLD - Control	LD - Test AB	LD - Test Slip	LD - Standard Slip	LD - Standard AB	HSD at 95%	Prob
Overall Degree of Difference	0.3 _b	3.2 _a	3.4 _a	3.7 _a	4.1 _a	4.5 _a	1.51	0.0001
Bitter	0.3 _b	2.1 _a	2.3 _a	2.4 _a	2.6 _a	3.0 _a	1.10	0.0001
Sweet	0.0	0.3	0.0	-0.1	0.0	0.4	0.94	0.5181
Sour	0.0 _b	1.0 _a	0.7 _{ab}	1.0 _a	0.8 _{ab}	1.3 _a	0.79	0.0011
Salty	0.0 _b	0.6 _a	0.3 _{ab}	0.4 _{ab}	0.6 _a	0.6 _a	0.51	0.0087
Oily mouth-feel	0.2	1.5	1.1	1.0	1.4	1.2	1.27	0.0513
Astringent	0.1 _b	2.1 _a	2.1 _a	2.2 _a	2.7 _a	2.6 _a	1.10	0.0001
Irritating	0.2 _b	1.8 _a	2.1 _a	2.1 _a	2.5 _a	2.6 _a	1.12	0.0001
Crayon	0.1 _b	1.5 _a	1.4 _a	1.6 _a	1.7 _a	1.6 _a	1.25	0.0052
Soapy	0.1 _b	1.8 _a	1.5 _a	1.9 _a	1.9 _a	2.1 _a	1.40	0.0010
Heated plastic	0.3 _b	3.1 _a	3.4 _a	3.1 _a	4.0 _a	3.9 _a	1.52	0.0001
Skunky	0.0 _b	0.9 _{ab}	0.9 _{ab}	1.3 _a	1.0 _{ab}	1.3 _a	1.14	0.0223
Chlorine	0.1 _b	1.2 _a	1.3 _a	1.1 _{ab}	1.5 _a	1.2 _a	1.03	0.0032
Vinegar	0.0	0.3	0.3	0.3	0.3	0.4	0.45	0.1817
Fruity	0.1 _c	1.3 _b	1.2 _b	0.9 _{bc}	1.2 _b	2.5 _a	0.96	0.0001
Earthy/Musty	0.1 _b	0.7 _{ab}	0.8 _{ab}	1.8 _a	1.4 _a	0.8 _{ab}	1.21	0.0039
Ranking (Friedman's)	1.3 _b	3.5 _a	3.6 _a	3.7 _a	4.3 _a	4.6 _a	NA	0.0001

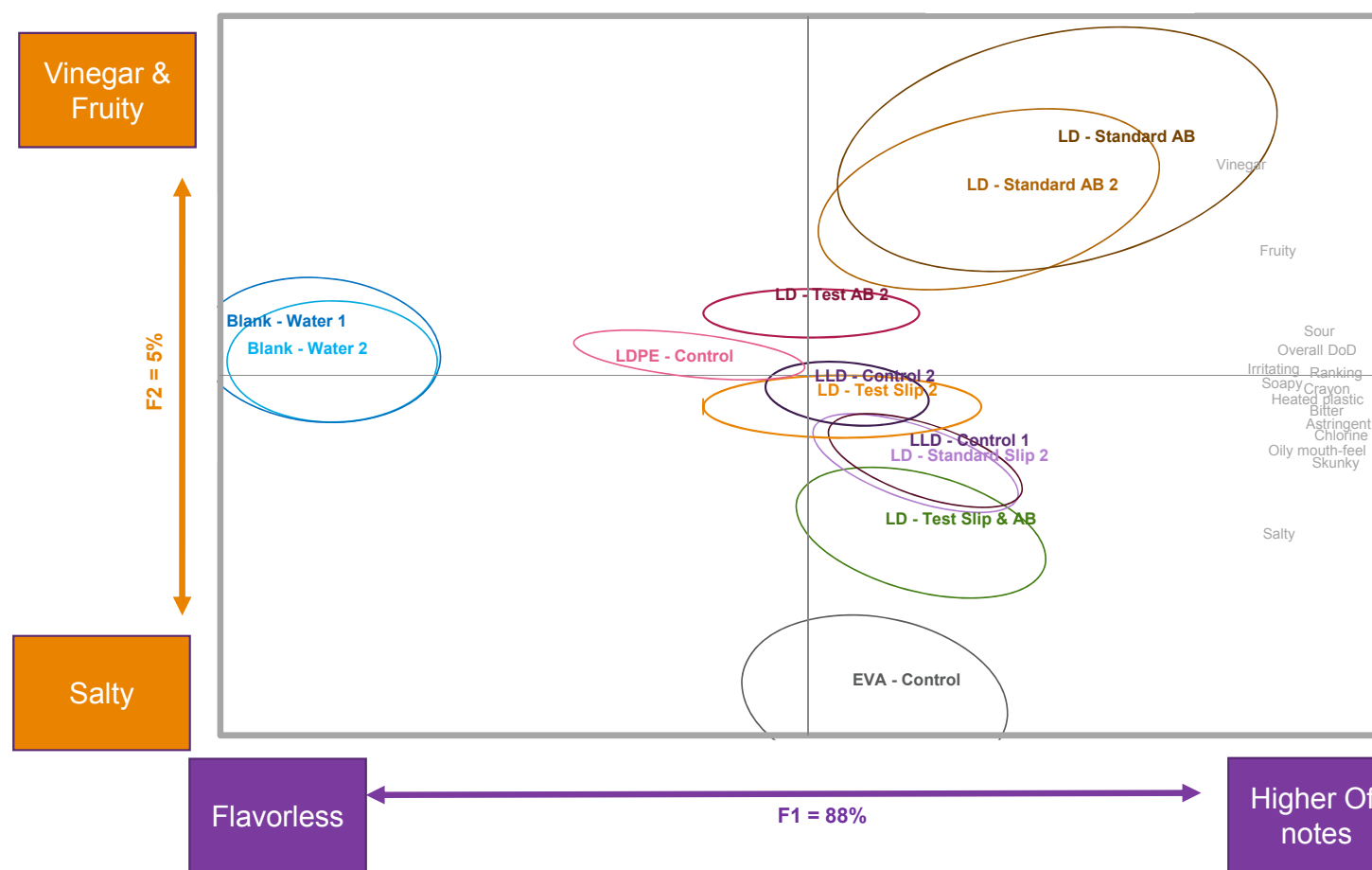
Within an attribute, products sharing a common letter are not significantly different at the 95% confidence level as tested by ANOVA with Tukey's HSD for Post Hoc comparisons.

Lowest in attribute	Not Sig. Different from Lowest	Not Sig. Different from Highest or Lowest	Not Sig. Different from Highest	Highest in attribute
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PCA of DoD Map for Additive Study - Set 2



PCA and DoD Map - All Samples in Additive Study



Confidence Ellipse
Product ellipses that do not intersect are different in the multivariate space.

STUDY 1 CONCLUSIONS AND OBSERVATIONS

1. Water stored in monolayer PE films had significantly higher flavor and attribute scores than control water. *Polyethylene Packaging materials, can change the sensory attributes of food.*
2. Slip and Antiblock Masterbatches can affect the sensory properties of water (and food products)
 - Samples with different slip and/or antiblock masterbatches had different attribute scores
3. Sensory scores showed good reproducibility of samples evaluated in different tests.
LLD Control and LD Standard AB samples delivered similar scores when tested among different samples.



STUDY 2 - Sensory Evaluation of Common LLDPE Sealants

OBJECTIVE: Measure and Compare the Sensory Properties of Water Stored in Monolayer Film Pouches made with Common LLDPE Sealant Resins.

Films For Study 2: Sensory Evaluations of Common LLDPE Sealants

Descriptor	Melt Index (g/10 min @ 190 C)	Density (g/cc)	Comonomer	Mfg. Process
Control - Octene	1.0	0.920	Octene	Solution
Octene 914	0.9	0.914	Octene	Solution – Multi
Butene 918	1.0	0.918	Butene	Gas Phase
Octene 920	1.0	0.922	Octene	Solution*
Plastomer 905 Blend with 50% Control	0.9	0.912	Octene	Solution – Multi
Plastomer 908	1.0	0.908	Octene	Solution – Multi
Plastomer 883 Blend with 50% Control	1.1	0.901	Octene	Solution*
Plastomer 902	3.0	0.902	Octene	Solution
Hexene 916	1.4	0.916	Hexene	Slurry Loop*
Octene 916	0.65	0.916	Octene	Solution Multi

Monolayer 3.5 mil Blown Films - Standard Conditions
 Very Thorough Die Cleaning and Purging Before Production
 Film Tubing converted into pouches within 72 hours

NOVA SCLAIR® FP120-A Control Used For Degree of Difference

LESS THAN CONTROL							Like Control	MORE THAN CONTROL						
Very Much Less	Much Less	Moderately to Much Less	Moderately Less	Slightly to Moderately Less	Slightly Less	Very Slightly Less	No Difference	Very Slightly More	Slightly More	Slightly to Moderately More	Moderately More	Moderately to Much More	Much More	Very Much More
-7	-6	-5	-4	-3	-2	-1	0	1	2	3	4	5	6	7

SLIP and Antiblock Study

Water Control 

Test Samples

LLDPE Sealant Study

 Control - Octene

Known
Control

Sample
A

Sample
B

Blind
control

Sample
C

Sample
D

Sample
E

Study 2 Samples Stored at 90° F for 28 days

Degree of Difference and PCA Scores - Sealant Study

Attributes	Plastomer 883 + 50% FP120-A	Butene 918	Blind Control Octene	Plastomer 908	Octene 914	Plastomer 902	Plastomer 905 + 50% FP120-A	Octene 920	Octene 916	Hexene 916	HSD at 95%	Prob
DoD Control	-2.1 f	-0.9 f	0.2 e	0.4 de	0.5 de	0.9 cd e	1.6 bcd	2.1 abc	2.5 ab	3.0 a	1.2	0.0001
Overall Aroma	0.9 f	1.2 e	1.4 de	1.4 cde	1.7 bc	1.4 de cd e	1.6 bcd	1.6 bcd	1.7 b	2.3 a	0.3	0.0001
Bitter	0.9 g	1.5 f	1.7 de	1.6 ef	1.9 cd	1.9 cd e	1.9 cde	2.1 bc	2.2 ab	2.4 a	0.3	0.0001
Sour	1.1 e	1.3 de	1.7 bc	1.5 cd	1.6 bc	1.6 bc	1.7 abc	1.8 ab	1.8 ab	1.9 a	0.3	0.0001
Oily mouth-feel/ Thickness	1.7 g	2.4 df	2.6 de f	2.6 cde f	2.9 ab	2.7 bc d	2.7 bcd	2.7 cde	2.9 abc	3.0 a	0.3	0.0001
Astringent	2.8 e	3.8 cd	3.9 bc	3.5 d	3.9 bc	3.7 cd	3.8 bc	4.0 bc	4.1 ab	4.2 a	0.3	0.0001
Irritating	0.8 e	1.4 d	1.7 cd	1.8 bcd	1.6 cd	1.8 bc d	2.0 bc	2.0 bc	2.2 ab	2.5 a	0.5	0.0001
Crayon	0.5 d	1.0 c	1.2 c	1.3 bc	1.2 bc	1.4 bc cd e	1.2 bc	1.5 b	1.5 b	2.0 a	0.4	0.0001
Soapy	0.0 f	0.2 f	0.5 de	0.3 ef	0.6 cd	0.6 cd e	1.1 ab	0.9 bc	1.0 b	1.4 a	0.4	0.0001
Heated plastic	1.6 e	2.6 d	3.1 c	2.9 cd	3.0 c	3.0 cd	3.1 c	3.8 b	4.7 a	4.6 a	0.4	0.0001
Chlorine	0.3 f	0.4 ef	0.6 de f	1.0 cd	1.2 c	0.9 cd	0.8 cde	1.6 b	1.8 b	2.6 a	0.4	0.0001
Earthy/Musty	0.2 c	0.9 b	0.9 b	1.1 ab	1.4 a	1.2 ab	1.0 ab	1.2 ab	1.4 a	1.4 a	0.4	0.0001

DoD and PCA Map of All Sealants – Both Sample Sets

Confidence Ellipse

Product ellipses that do not intersect are different in the multivariate space.

F2 = 5%



Flavorless

F1 = 88%

Higher Overall
Aroma, Off Notes,
Irritating, Oily
Mouthfeel, Thickness



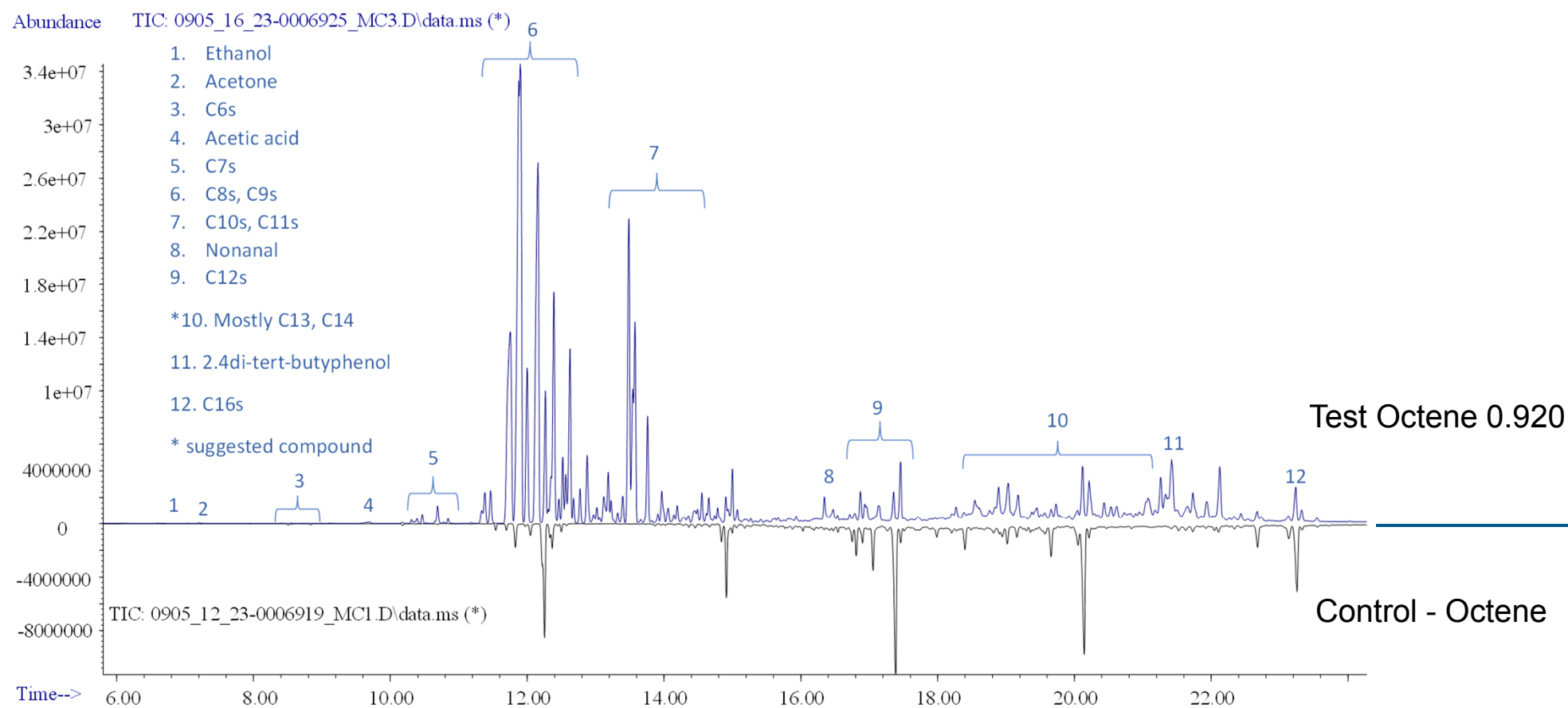
Investigation 3 – Chemical Sources of off-Flavor and Attributes

OBJECTIVE: Correlate Specific Sensory scores and differences to specific types and levels of chemicals in polyethylene sealants

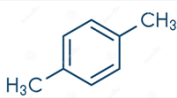
Can Sensory and Attribute Scores be Correlated with Specific Compounds in the Sealant?

Can Sensory Scores be Improved by Lowering the Concentration of Specific Compounds?

Total Volatile Organic Compounds (VOCs) as a Sensory Indicator



Odor Detection Thresholds for C2 Chemicals

Chemical Compound	Odor Detection Threshold (50 th percentile)	Possible Packaging Source	Molecule / Chemistry
Ethane (Most Olefins)	"Odorless"	Polyethylene oligomers	CH ₃ – CH ₃ CH ₃ – (CH ₂ – CH ₂) _n – CH ₃
Ethanol	80 ppm	Additives, various	CH ₃ – CH ₂ – OH
Xylene (Aromatics)	1.0 ppm	Various	
Acetic Acid	0.75 ppm	Vinyl Acetate Degraded PE	CH ₃ – C(=O) – OH
Acetaldehyde	0.05 ppm	PE Oxidation	CH ₃ – CH = O
Ethylamine	0.27 ppm		CH ₃ – CH ₂ – NH ₂
Ethanethiol	2.8 <u>ppb</u>		CH ₃ – CH ₂ – SH

“Functional Groups” change the flavor profile.
(Not the pure polyethylene)

Flavor Characteristics of Specific Chemicals

Compound	odour description (PubCHEM, NIH)	Bitter	Sour	Oily Mouthfeel/ Thickness	Astringent	Irritating	Crayon	Soapy	Heated plastic	Chlorine	Earthy/ Musty
Benzenamine, N,N-dimethyl-	amine-like, fishy					x					
1-Nonanol	rose, fruity, floral, citronella										
1-Decene	pleasant										
Nonanal	rose-orange, floral, fruity, waxy, green						x				x
Octanoic acid	mild, unpleasant, fruity-acid	x	x		x	x					
2-Nonanone	fruity, floral, fatty, herbaceous			x							x
Estragole	anise	x									x
α -Terpineol	floral, lilac										
Octanoic acid, ethyl ester	wine, brandy, fruity floral		x		x						
Acetophenone	sweet, pungent, strong medicinal, almond-like, resembling oranges					x				x	
3-Decanone	citrus, orange, floral, slightly fatty			x							
1-Tetradecene	mild, pleasant										
Nonanoic acid	slight, fatty, coconut			x							
Decanal	floral-fatty, citrus, orange-peel, Penetrating, sweet, waxy, floral, citrus, pronounced fatty odor that develops a floral character on dilution			x		x	x	x			
ketone	fruity, floral, fatty, herbaceous			x							x
1-Octadecanol	faint										
Benzothiazole	quinoline-like, rubbery, unpleasant						x				x
Naphthalene, 1-methyl-	Coal tar or mothball, earthy, phenolic					x					x
1-Decanol, 2-hexyl-	mild sweet										
2-Undecanone	citrus, fatty, rue-like, strong			x		x					
polyethylene glycol	mild										

Chemical Groups Detected by GC x GC

Compound	Control Octene	Octene 914	Octene 920	Plastomer 905 + FP120 BL	Plastomer 883 + FP120 BL	Plastomer 908	Butene 918	Octene 916	Hexene 916	Plastomer 902	odour description (PubCHEM, NIH)
Groups											
Isoparaffins - Total	5.67E+03				5.03E+03		5.18E+03	5.04E+03		5.49E+03	No Odor (Mouth Feel?)
Arsine - Total	7.74E+04				2.05E+04		2.11E+04	5.40E+04	8.11E+04	2.30E+04	Disagreeable garlic
C10+ alkene - Total	6.94E+08	6.29E+08	2.86E+08	2.93E+08	4.64E+08	7.37E+08	2.53E+08	2.48E+08	4.67E+08	3.36E+08	fuel-like
Diterpenoids - Total	1.25E+05	8.30E+07				1.31E+08		3.14E+06			Various, Floral
`			4.72E+05		1.14E+05						faint, sweet
Thiols - Total	1.68E+08	1.83E+08	4.99E+08	5.50E+07	8.28E+07	1.59E+08	7.25E+07	7.90E+07		1.62E+08	(generally thiols have strong odours resembling garlic)
Peak Intensity				Low			Medium		High		

➤ 400 distinct molecules or chemical groups

What chemicals or chemistries are present in worst, absent in best?

Conclusions

1. The sensory profiles of sealants and additives are important performance criteria that should be considered when selecting materials for sensitive food products.
2. Sensory test methods for packaging should be optimized to provide sample differentiation and the most useful information. Pre-screening materials by trained panel leaders is recommended to ensure samples, preparation and test methods will provide useful information.
3. The specific components in Additive Masterbatches can significantly affect sensory properties of packaged food products
4. Polyethylene sealants can impart a wide range of flavors. The type and level of flavors are mostly attributable to functional ingredients and thermo-oxidative by-products.

Acknowledgements

NOVA Chemicals : Andrea Rangel, Krista Stang, Tracy Li, and Sierra Ross
SENSATION RESEARCH



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