CoolZoom® Flavors: One Year Shelf Stability Analysis

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Abstract:
The retention and stability of three commercial flavor powders produced using a novel low temperature drying process are reported. The flavors were formulated using a high solids approach and dried in a production dryer at an inlet temperature of 110 °F (43 °C), considerably lower than traditional spray dry practices. The gas chromatography/mass spectrometry ("GC/MS") analysis of the resulting powders show high flavor retention ranging from 93% to 98%. Triangle test results comparing recent and 1-year old samples showed no distinguishable difference for Chocolate and Lemon flavors and a very slight difference in Fruit Punch flavor during storage. Oxidation over a 12-month period was analyzed as well showing an evolution of less than 1% of the total retained flavor. These results illustrate the significant retention and stability improvements that result from the Zooming® low-temperature spray drying process.

Introduction:
Flavors are complex formulations of aromatic molecules, such that when combined properly deliver consumer preferred aroma and taste. Many of these molecules are extremely volatile in nature and traditional spray drying has caused the evaporation of important aromatic components followed by significant oxidation over time. A patented[1] low-temperature spray drying process known as Zooming® enables extremely high retention and exceptional stability of volatile flavor compounds in CoolZoom® flavors. Reineccius and Coulter[2] compiled a large review of methods to improve the retention of compounds in flavor powders encompassing emulsion droplet size, viscosity, particle size, particle composition, dryer inlet and outlet conditions among others. CoolZoom® flavors deliver larger fully dense particles utilizing carrier systems that promote retention all enabled by the Zooming® low temperature spray drying process. In another review, Reineccius[2] highlighted the complex nature of stability in flavor powders. Below we will discuss and highlight how the characteristics of the CoolZoom® flavors and temperature of the production process impact flavor stability.

Particle Size
The effects of particle size on oxidation were reported by Sootitantiwat et.al.[3] finding a 63% decrease in limonene oxide generation by increasing the particle size from 27 to 60 microns. This effect was explained in terms of the smaller specific surface area for the larger diameter powder which decreased from 0.68 microns (small particle) to 0.12 microns (large particle). The smaller specific surface area decreases the effective surface area for the d-limonene to release or react. The CoolZoom® flavors in this study have a mean particle size of 86-88 microns, significantly larger than traditional spray dried particles.

Particle Composition
The composition of a particle consists of several elements including carrier materials, emulsifiers, and flavor components. Studying the encapsulation and protection of volatile compounds, Kanakande[4] found for cumin that

the use of gum arabic or gum arabic and modified food starch/maltodextrin blends gave the greatest protection of encapsulated ingredients. Shiga et.al.[5] was able to demonstrate improved stability in flaxseed oil by reducing the emulsion droplet size and subsequently surface oil. A similar study by Soottitantawat et.al.[6] also showed the effect of decreasing emulsion droplet size on improving the retention of d-limonene, ethyl butyrate and ethyl propionate model compounds. The CoolZoom® flavors utilize these types of carrier systems to promote stability. At the same time, the emulsion droplet sizes achieved in the Zooming® process reach 1 micron or less, further promoting flavor stability.

Particle Structural Evolution

CoolZoom® particles with high solids composition produced at significantly lower temperature exhibit a more homogenous microstructure. The processes leading to solid particle formation is complex, having many facets as discussed in a review by Sadek et.al.[7] Senoussi et.al.[8] found that retention of citral and linalool increased 12% when the dry matter increased from 45% to 50%. The ZoomEssence emulsion process utilizes low water content resulting in higher solids to oil ratios and subsequent increased viscosity. See reference 1 for further discussion.

Utilizing a high solids emulsion process, CoolZoom® flavors exhibit a dense matrix and enhanced morphology that protects actives during storage and food processing, allowing for efficient delivery of active flavor components. The dense nature of the CoolZoom® particles is illustrated in the cross-sectional scanning electron micrograph in **Figure 1**. The high solids emulsion coupled with low temperature drying minimizes shrinkage during the drying process eliminating the hollow core microstructure of traditionally dried particles.

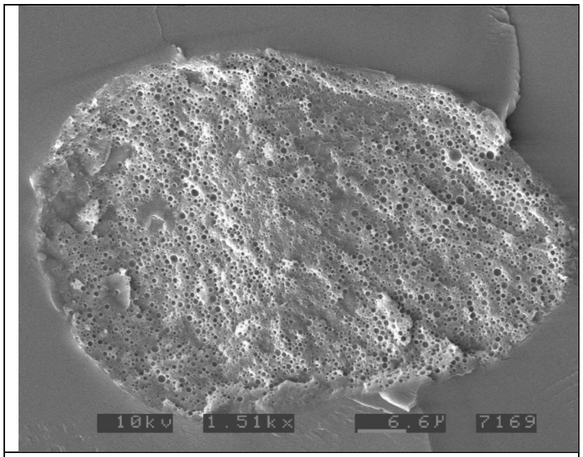


Figure 1. SEM cross-section image of a representative low temperature dried particle with a 20% oil load, illustrating the fully dense structure.

Drying Temperature

The formation of powders from liquid precursors by high temperature spray drying has been in existence for over a century. During the last 50 years, a significant body of literature has been generated focusing on how flavor powder stability is impacted by the drying temperature. Rulkens and Thijssen[9] showed that volatile retention was predominantly determined by the diffusion coefficient of the volatiles in the drying drops. Retention increased with increasing dissolved solids and highest retentions were obtained at the lowest air inlet temperature. Effects of spray drying temperature on drying of roselle (hibiscus sabdariffa) extracts showed a significant increase in the generation of degradation compounds as drying temperature increased from 150°C to 210°C.[10]

ZoomEssence® uses patented low-temperature spray drying technology known as Zooming® to retain delicate actives by avoiding evaporative losses and thermal degradation. The Zooming® process spray dries liquids at approximately 43°C, an unprecedented low operating temperature of spray drying at commercial production rates.

In order to understand the stability properties of CoolZoom® flavors a study was carried out combining both GCMS and sensory information. In this approach, it is possible to identify compound evolutions that may contribute to shorter shelf life. This evaluation was performed on chocolate, fruit punch and lemon flavors, which encompass a significant portion of the flavor market. The results of these studies are presented here.

Materials and Methods:

We chose three CoolZoom® flavors that are routinely manufactured for commercial use. The samples evaluated are listed in **Table 1**. The flavors were manufactured on the same equipment with substantially similar operating parameters. Samples were obtained from the Quality department, thus being kept in 4 ounce high-density polyethylene jars with no exposure to light and stored between 65˚ and 85˚ Fahrenheit.

Flavor samples were weighed out into 50-mL culture tubes and extracted for component analysis. Components were identified by mass spectrometry and quantified by flame ionization detector. Particle size was measured on a Beckman Coulter LS 13 320 laser diffraction particle sizer and reported using a volumetric distribution.

Table 1. Flavors analyzed in this study

CoolZoom® Flavor	Manufacture Date
Chocolate	11/17/17
Chocolate	11/15/18
Fruit Punch	12/18/17
Fruit Punch	12/21/18
Lemon	12/4/17
Lemon	12/10/18

Sensory Protocol

ASTM (E1884-04) triangle tests were conducted with a panel of 24 people. This number was chosen to reduce the risk of incorrect different-perception and non-different-perception to 5% while maintaining a differentiation threshold of 50% of the panelists. A set of samples with clear differentiation occurs at 50% of panelists differentiating the two samples. Identical samples are defined when 33% or less of panelists can differentiate between the two samples. In a triangle format, this is consistent with random guessing for evaluation. Sample sets are prepared so that a total of 6 separate combinations of samples (ABB, BAB, BBA, BAA, ABA and AAB) are presented to the panelists. Each panelist is given a set of samples, an assessment card, are informed 2 of the samples are like and are asked to identify the non-like sample. Results are then tallied and assessed by the moderator. Panelists used were employees of ZoomEssence Inc. between the age of 23 and 71 years of age with typical taste and smell functions.

Results and Discussion:

Chocolate, Fruit Punch and Lemon samples were chosen for this evaluation due to their high volume in the food and beverage market. Additionally, this set covers non-volatile, volatile, natural extract and compounded flavors. The initial set of retains were characterized prior to sensory and GC/MS assessment.

The products have reproducible production characteristics. Mean particle size for all products measured is between 86 and 88 microns, which is representative of products manufactured via the Zooming® process. Particle size distributions are reported in **Figure 2**, showing the reproducibility of the processing conditions between different products.

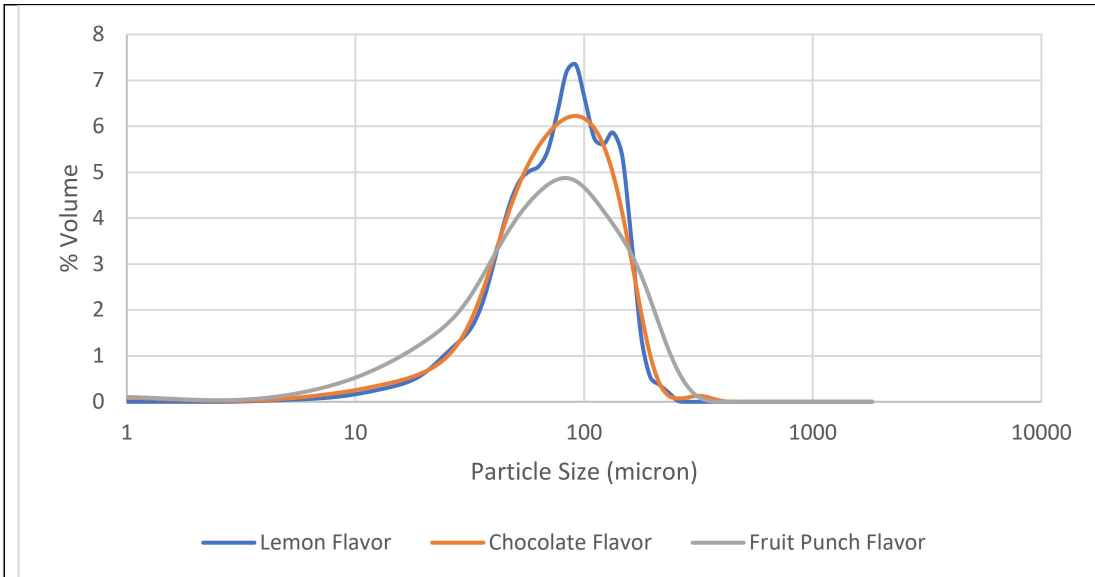


Figure 2. Particle size distibrutions for Lemon, Chocolate and Fruit Punch flavors, reported on a volumetric basis.

Sensory Evaluation:

A 24-person panel was used to evaluate 1 year old and recently manufactured retains of the chosen products. Values are reported in **Table 2.** The proportion of correct (Pc) responses, identifying the unlike sample in the triangle, was 33% for chocolate and lemon samples and 42% for fruit punch samples. The proportion of the population that would be able to distinguish between the samples (Pd) is thus determined from the Pc by the following equation:

$$Pd = 1.5 * Pc - 0.5$$

The Pd values for chocolate and lemon are 0%, considering Pc was no higher than random guessing (1/3) in a triangle panel. The Pd for fruit punch was 13%, indicating a small proportion of the population may be able to discriminate between 1 year old and recent lots of product. According to ASTM E1885-04 19.2.3.1, Pd values less than 25% represent a small percentage of distinguishers. Thus, the values indicate a small proportion of the population are capable of distinguishing between recent and 1-year old samples of CoolZoom® flavor.

Table 2. Proportion correct (Pc) and proportion distinguishing actual (Pd actual) with 90% confidence bounds for discriminatory triangle evaluation.

	Chocolate	Fruit Punch	Lemon
Proportion Correct	33%	42%	33%
Lower 90% Confidence	0%	0%	0%
Pd Actual	0%	13%	0%
Upper 90% Confidence	18.5%	31.8%	18.5%

GC/MS Analysis:

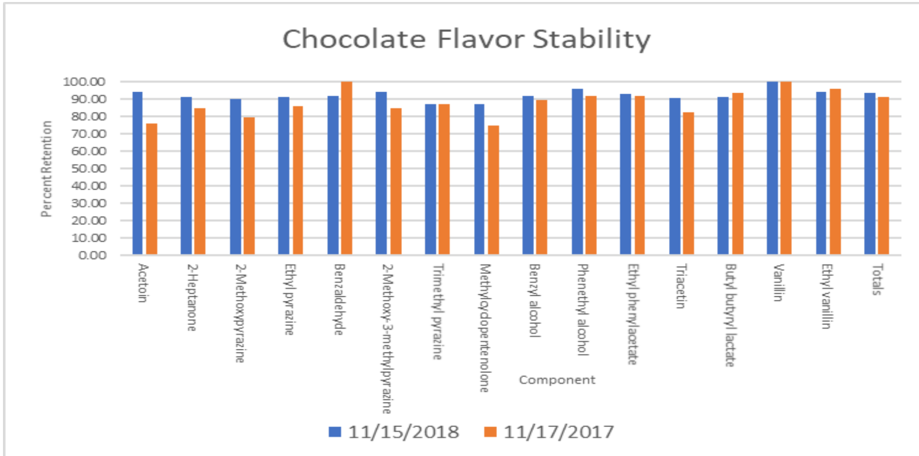
The retention of flavor in the powder was measured via solvent extraction. All flavors were retained in excess of 90%. A triplicate measurement was performed on the fruit punch samples indicating the standard deviation on the measurement method was within 2%. The 93% retention rate of the chocolate flavor could be attributable to the water soluble components associated with chocolate flavors, which are difficult to account for in solvent based extractions. Retention for fruit punch and lemon flavors was exceptionally high at 96.4% and 98%. Data is reported in **Table 3**.

Table 3. Percent retention of flavor within chocolate, fruit punch and lemon samples as measured in the recent retain.

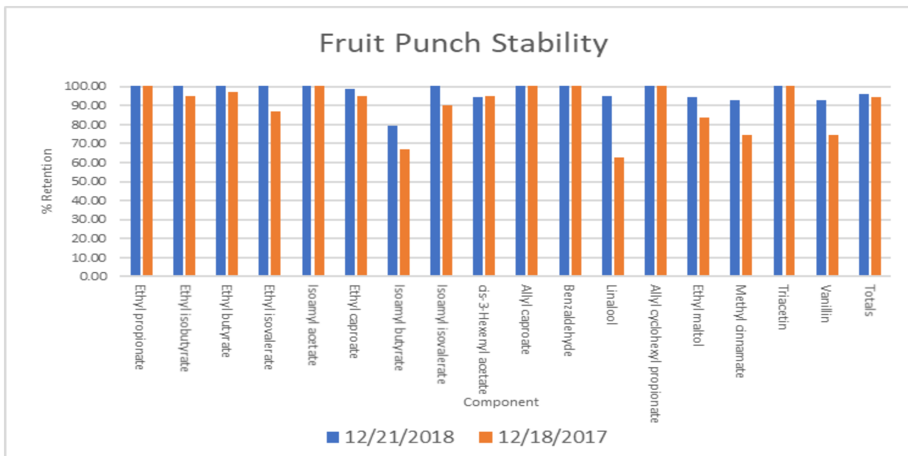
	Chocolate	Fruit Punch	Lemon
% Total Retention	93%	96.4 ± 1.7%	98%

The percent retention from GC/MS measurements for the three flavors studied are shown in **Figures 5a, 5b and 5c** on the following page.

5a)



5b)



5c)

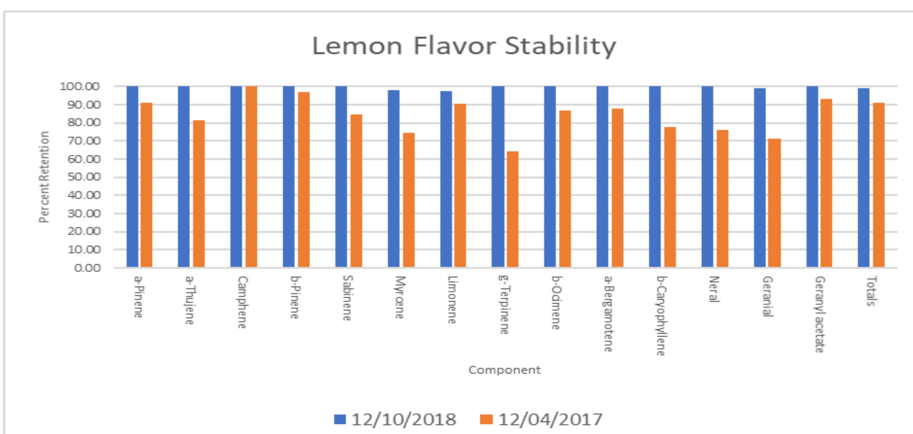


Figure 5. Percent retention for a) Chocolate b) Fruit Punch and c) Lemon Flavor showing results for two measurements made on the same sample taken nearly one year apart.

The oxide evolution was measured in the samples and reported in **Figure 6.** Limonene is the primary component of lemon flavor and is a component of the fruit punch flavor. In the presence of oxygen, this converts into limonene oxide, carveol and carvone. When the percent oxides is measured, the amount of limonene oxide, carveol and carvone are summed and divided by the amount of limonene. No oxides were seen in the recent lemon sample and over 12 months of storage a very modest 0.93% evolved. In the fruit punch, the limonene raw material had an appreciable amount of limonene oxide present and this increased during the storage life. Within the chocolate, limonene was not present and therefore no oxides were measured. The complete results are summarized in **Table 4.**

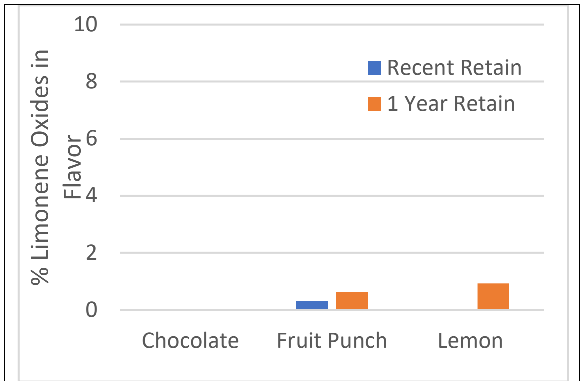


Figure 6. Percent limonene oxides in the flavor.

Table 4. Percent retention of flavor within chocolate, fruit punch and lemon samples as measured in the recent retain.

	Chocolate	Fruit Punch	Lemon
% Retention	93%	96.4±1.7%	98%
% Limonene Oxides after 1 Year	0%	0.62%	0.93%
% Population Distinguishing Samples	0%	13%	0%

Conclusion

The 1-year shelf stability claim of CoolZoom® flavors has been confirmed with both discriminatory triangle testing and GC/MS. An ASTM triangle discrimination test was designed for 95% with a 50% proportion of distinguishers thus requiring 24 panelists. Triangle test results comparing recent and 1-year old samples procured from quality retains showed no distinguishable difference for Chocolate and Lemon flavors during aging. There was a small distinguishable difference (13%) for the Fruit Punch flavor. GC/MS analysis of the retains showed flavor retention greater than 95% for Fruit Punch and Lemon flavors and 93% in a Chocolate flavor. Oxidation over the 12-month period was analyzed as well showing oxidation evolution less than 1% of the total retained flavor.

References

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