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SOLUBLE COFFEE: SHELF LIFE STUDIES

INTRODUCTION

SOLUBLE COFFEE is the dried water extract of roasted ground coffee. The product consists of brown colored free-flowing particles of uniform size (Moores and Stefanucci, 1964). Soluble coffee comprises two distinct product types, viz., spray dried and freeze dried. Soluble coffee producers add natural coffee aroma in coffee oil to the powder. According to Federal Specification Coffee, Instant (1964 Amend. 1967) the requirements for spray-dried coffee are: Moisture content 3% maximum, and caffeine 3.2% minimum. The distinctive features of freeze-dried coffee as cited in the military Interim Purchase Description S-11-2 (1972) are: moisture content 2.6% maximum; caffeine 2.2% minimum; and carbohydrates 35% maximum on dry basis. In general the same compounds found in fresh coffee are contained in soluble coffee; however, the ratios and amounts are completely different and are dependent upon the process of removing water (Gianturco, 1967). As more highly volatile and chemically unstable flavor constituents are retained or added to soluble coffee, the problem of stability, i.e., staling, increases.

Deterioration of initial quality or staling of these coffees has long been attributed to reactions with headspace oxygen. Sivetz and Foote (1963) claimed that flavor changes in properly packed soluble coffee would be nominal after a period of 6 months to several years if it were sealed air-tight and stored at less than 21°C. Furthermore they stated that instant coffee stability in air storage when not aromatized (coffee oil add-back) was very good. Recent studies by Bishov et al. (1971) on a variety of freeze-dried foods stored under "zero" oxygen indicated a preference for these foods. Published shelf studies dealing with soluble coffees stored under "zero" oxygen headspace conditions were virtually nonexistant. In fact Wilhelm (1958a, b) in two articles reported about packaging requirements of instant coffee but he mainly discussed early work with commercial equipment and containers. White (1959) recommended using air tight containers to protect the quality of soluble coffee but also did not offer supportive data by panel studies as to why low oxygen in the headspace of the packaged product was necessary. The quality of soluble coffee is mainly due to the volatile aroma of over 300 compounds with boiling points varying between 160°C and over 250°C (Thijssen 1969). The instability of the aroma flavor of soluble coffee during storage is the major problem facing the coffee industry. Loss of flavor due to aging has been reduced somewhat by better packaging, by more efficient distribution, and rapid delivery and by more care in stock rotation and turnover (Lee, 1965).

Elder (1940) reported that roasting coffee beans resulted in increasing stability of the coffee oil due to formation of heterocyclic imino compounds, some of which showed antioxidant properties. The oxygen of air is potent enough to oxidize important constituents of flavorless compounds (Lee, 1965).

This study was undertaken because the military is procuring freeze-dried coffee in limited quantities and would like to design a system that offers adequate protection against deterioration. A study of this nature could entail a multitude of research; however, it was limited in scope to answer practical questions such as: does a low oxygen headspace protect these coffees? How does a freeze-dried coffee compare with a spray-dried coffee as to keeping quality stored in various packs, and is a definite maximum tolerance on headspace oxygen needed in the purchase document or specification? Currently a residual oxygen of not greater than 2% is a customary specification standard for storing freeze-dried foods (U.S. Army Natick Laboratories, 1967). According to Palese (1972), a low oxygen atmosphere pack of this nature is costly (about 2¢ per 8-oz pack).

EXPERIMENTAL

Materials

Spray-dried soluble coffee. A fresh batch of spray-dried agglomerated soluble coffee was procured from a coffee producer in their commercial 10-oz jars. The moisture content was 4.5%.

Freeze-dried soluble coffee. A fresh batch of freeze-dried soluble coffee was procured from a coffee producer in their commercial 8-oz jars. The moisture content was 2.0%.

Gases

One gas was a mixture of analytical grade 98% nitrogen/2% oxygen v/v. The other gas used was 5% hydrogen v/v in nitrogen. Catalyst was palladium powdered metal, 0.5% w/w on kaolin as pellets.

Packaging

8 oz each of both spray-dried instant and freeze-dried instant soluble coffees were repacked in hermetically-sealed 401 × 411 cans in a controlled humidity room regulated at 21°C and 20% relative humidity.

For the air pack no gas was added to the headspace. To achieve 2% oxygen, the contents of the can were purged four times in a closed chamber by pulling a 30-in. vacuum holding 30 sec to remove any atmospheric gases and then back flushing from a tank the desired 2% oxygen mixture gas. For the "zero" oxygen packs the same procedure as above was followed except that the cans contained 0.5g of a palladium catalyst (Houdry Processing & Development Co.) wrapped in "Kimwipes" tissue paper and fastened to the can lid with "Scotch" transparent tape and the cans purged with the gas containing 5% hydrogen/95% nitrogen. The cans after closure were dipped in molten paraffin wax, a treatment found necessary to prevent gas leakage through the can lid double seam seal closure. All cans were stored at 37.8°C and withdrawn after 1 wk, 1 month, 2 months, 3 months, 4 months, 6 months, 9 months and 12 months for headspace gas analysis and sensory evaluation. The samples were run in duplicate.

Headspace gas analysis

Residual oxygen, carbon dioxide and residual hydrogen contents were determined by the gas chromatographic method of Bishov and Henick (1966). Sampling was achieved with a can-lid piercing needle on a gas-tight syringe. The sampling area on the can lid was first covered with a self-adhesive tape of closed-cell polyurethane foam. RTV sealant was used to cover the external patch to prevent leakage before sensory evaluation.

Panel testing

Two different panels were used to evaluate the stored samples.

A technological panel of 12 selected judges who reported judgements on color, flavor, odor and appearance on a 9-point scale (Peryam and Shapiro, 1955). 40g of spray-dried soluble coffee were dissolved in 1 gal of boiling water and served as black coffee in 2-fl oz porcelain cups at a temperature of 63°C. 36g of freeze-dried soluble coffee were reconstituted in a gallon of boiling water and served in the same manner as the spray-dried soluble coffees. Spray-dried or freeze-dried coffee treatments of air pack, 2% oxygen pack and "zero" oxygen pack were served to the technical panel in separate sessions as a 3 of 3 complete block design.

A consumer panel of 30 judges randomly selected at each session reported preferences on a 9-point scale (Pilgrim and Peryam, 1958). The panel was served coffee treatments initially and after 12 months.

RESULTS & DISCUSSION

Headspace gas studies— Soluble coffees

Data (Table 1) indicated that as the soluble coffees aged, there was a general decrease in the amount of oxygen in the headspace gas of both air and 2% oxygen packs of spray-dried soluble (SDS) and freeze-dried soluble coffees (FDS). In the "zero" oxygen packs there was no appreciable change in the oxygen content over the entire storage period. This suggested that the cans were not leaking and that the coffee was not absorbing oxygen. To check this, the amount of residual hydrogen that remained in the "zero" oxygen packs was analyzed. The headspace gas analysis indicated the presence of hydrogen in the gas phase, thus confirming the absence of a gas leak and that all of the oxygen absorbed during processing on the coffee particles reacted with the hydrogen resulting in its complete absence in the gas phase.

Samples packed in "zero" oxygen atmosphere showed no significant difference in oxygen content yet soluble coffee packed in either air or 2% oxygen atmosphere showed a gradual decrease in oxygen headspace content during storage. For example, the SDS coffee absorbed more oxygen than the FDS coffee. Comparing the two coffees as to carbon dioxide evolution, it was found that SDS coffee gave off significantly more carbon dioxide (air and 2% oxygen packs) than FDS coffee stored under similar conditions. Conversely, at "zero" oxygen there was no difference in carbon dioxide evolution between these two coffees.

Generally speaking, SDS instant coffee

Table 1—Headspace oxygen and CO_2 for soluble coffees[a] packed in "zero" oxygen, 2% oxygen and air in hermetically-sealed containers stored at 37.8°C

Sample	% Initial O_2	CO_2	1 week O_2	CO_2	1 month O_2	CO_2	3 months O_2	CO_2	4 months O_2	CO_2	6 months O_2	CO_2	9 months O_2	CO_2	12 months O_2	CO_2	sig. O_2	sig. CO_2
SDS air	20	1.0	17.5	1.9	13.6	6.7	7.8	14.7	6.5	15.5	4.1	18.5	—	—	—	—	NS	*
FDS air	20	1.0	14.2	1.2	17.3	3.0	9.7	6.3	7.9	7.4	4.4	8.5	2.8	8.9	—	—		
SDS 2%O_2	2	1.1	1.8	1.1	1.6	3.9	0.5	8.2	0.3	10.2	0.3	10.7	0.3	14.2	0.0	16.3	*	*
FDS 2%O_2	2	0.7	1.8	0.7	1.4	1.9	0.4	3.8	0.2	4.6	0.2	5.5	0.3	6.5	0.0	8.2		
SDS 0%	0	1.0	0	0.6	0.2	1.7	0.0	9.0	0.0	6.0	0.0	11.9	0.4	13.8	0.1	17.8	NS	NS
FDS 0%	0	1.2	0	1.4	0.1	4.8	0.0	3.6	0.0	4.1	0.0	5.5	0.3	6.4	0.0	7.7		

* Significant difference at 5% level
[a] SDS = spray-dried soluble; FDS = freeze-dried soluble; data mean of two samples

Table 2—Soluble coffees—Comparison of headspace gas treatments as judged by a technical panel using mean sensory ratings at each withdrawal from storage at 37.8°C[a]

Treatment		Init.	sig.	1 wk	sig.	1 mo	sig.	2 mos	sig.	3 mos	sig.	4 mos	sig.	6 mos	sig.	9 mos	sig.	12 mos	sig.
										Spray-dried soluble									
"Zero" oxygen	Color	7.0		6.8		7.4		7.1		7.1		7.3		6.9		6.8		7.2	
	Odor	6.3		6.7*		6.7		6.9*		6.5		6.0		6.2		6.0		6.1	
	Flavor	5.5		5.8		6.5		6.9		5.9		6.1		6.4		6.0		5.5	
	Appearance	6.9		6.8		7.3		7.1		7.0		6.8		6.9		6.7		6.9	
2% Oxygen	Color	7.0		6.9		7.1		7.1		7.2		7.3		6.8		6.9		7.1	
	Odor	6.3		6.1		6.3		6.4		6.5		5.8		6.1		6.3		6.2	
	Flavor	5.5	NS	5.4	NS	6.0	NS	6.1	NS	6.3	NS	5.3	NS	6.3	NS	6.4	NS	5.7	NS
	Appearance	6.9		6.8		6.9		6.9		6.9		6.8		6.7		6.8		6.8	
Airpack	Color	7.0		6.8		7.3		7.0		7.2		7.2		6.9		6.8		7.0	
	Odor	6.3		6.2		6.4		6.4		6.4		5.7		6.0		6.2		6.0	
	Flavor	5.5		5.5		5.8		6.4		6.3		5.3		5.8		5.3*		5.3	
	Appearance	6.9		6.8		7.2		6.8		7.1		6.8		6.6		6.4		6.6	
										Freeze-dried soluble									
"Zero" oxygen	Color	7.0		6.9		7.2		7.0		7.2		7.4		7.2		7.0		7.3	
	Odor	6.3		6.3		6.3		6.3		5.5		5.7		6.3		6.3		6.0	
	Flavor	5.8		6.0		6.3		5.9		6.3*		6.3		6.2		6.0		5.3	
	Appearance	6.9		6.8		6.9		6.8		7.2		7.3		7.1		7.0		7.1	
2% oxygen	Color	7.0		6.8		7.3		7.1		7.2		7.4		7.2		7.0		7.3	
	Odor	6.3	NS[2]	6.2	NS	6.4	NS	5.9	NS	5.7	NS	6.2	NS	5.8	NS	6.5	NS	6.5	NS
	Flavor	5.8		5.5		6.7		6.0		5.4		6.3		5.8		6.3		5.7	
	Appearance	6.9		6.8		7.3		6.8		7.3		7.4		7.3		7.0		7.1	
Airpack	Color	7.0		6.9		7.4		7.0		7.3		7.4		7.2		6.9		7.3	
	Odor	6.3		6.3		6.5		5.9		5.3		6.3		5.6		6.1		6.0	
	Flavor	5.8		5.9		6.5		5.7		5.4		6.4		5.3		6.1		5.5	
	Appearance	6.9		6.8		7.3		6.8		7.3		7.4		7.1		6.9		7.0	

[a] 5.0 = fair; 7.0 = good
* Significant 5% level; NS = not significant

produced greater quantities of carbon dioxide than FDS. Charting peak levels versus storage time, it was found that the two coffees varied as to when the highest level occurred but it was after 6 months storage at 37.8°C for the air pack of SDS, after 12 months for SDS 2% oxygen pack, and after 12 months for the SDS "zero" oxygen pack. For FDS the peak carbon dioxide level occurred at 9 months for the air pack, 12 months for the 2% oxygen pack and at 12 months at the "zero" oxygen pack. It should be emphasized that there were no samples available to analyze for headspace gases for the 9 month SDS air withdrawal or SDS 12 months of FDS samples in air pack. Furthermore the data in Table 1 indicate that as coffee ages at 37.8°C, headspace oxygen content decreased gradually and carbon dioxide content increased. In air packs where more oxygen was available absorption of oxygen and evolution of carbon dioxide by soluble coffees do not follow a direct 1:1 relationship. This increased level of carbon dioxide may originate from the carboxyl groups of the amino acids by nonenzymatic browning (Reynolds, 1963). Lockhart (1969) reported that roasted coffee contains 11–14% protein, 12–14% fat and 0.4–2.0% sugar. In an aqueous extract of roasted coffee the following amino acids were identified and quantified (mg/100g) dry wt: alanine (154), aspartic (172), glutamic (138), glycine (92), leucine (51), phenylalanine (103) and valine (154) (Underwood and Deatherage, 1952). The presence of sulfur-containing amino acids, cystine and methionine in aqueous extracts of roasted coffee had been reported by Barbera (1956).

Sensory panel tests

In Table 2 soluble coffees were evaluated by a technical panel at the same withdrawal time as when headspace gas analysis was performed. Data indicated that the odor of the spray-dried soluble coffee ("aromatized") was affected to some extent by oxygen in the headspace. The "zero" oxygen coffee samples were judged to be significantly better than either the 2% oxygen or air-pack treatments after 1 wk and after 2 months storage.

None of the other coffee treatments was judged significantly different as to odor, color, flavor or appearance when comparing like parameters at each withdrawal period until after 9 months at 37.8°C. At that time the flavor of the spray-dried coffee in the air pack was significantly different from the other packs. After 2 months at 37.8°C both SDS and FDS coffee developed an unusual shellac-like odor stored under "zero" oxygen. This odor was detectable by "smell" on opening the can. Furthermore, this odor persisted in the "zero" oxygen packs in subsequent withdrawal samples of these dried coffees. This odor was not apparent in any other treatments. Also, it did not carry-over into the reconstituted products of "zero" oxygen samples of black coffee and apparently had little or no effect on panel ratings. This aroma transformation may be due to the development of carbonyl compounds (Forss et al., 1967).

For freeze-dried soluble coffee, there were no significant differences for color, odor, flavor or appearance at any withdrawal period up to 1 yr except after 3 months at 37.8°C. At this withdrawal, the coffee stored under "zero" oxygen headspace was judged to be significantly better than the 2% and air packs. Since a technical panel is mainly concerned with quantitative differences in coffee quality but not its acceptance, the coffee samples were evaluated by a consumer panel. In Table 3 the data show that there were no differences between the spray-dried soluble coffee initially but after 12 months at 37.8°C, the spray-dried soluble coffee stored in 2% oxygen packs was judged by the panel to be significantly better in acceptance than the air pack, but not better than the "zero" oxygen or unstored new sample. A new sample was introduced to see what effect aged coffee had on consumer acceptance. Paradoxically, the 2% oxygen sample tasted as good as the fresh sample (control). For the freeze-dried coffee treatments the panel did not find a significant difference in acceptance for any of the headspace treatments. These data suggested that freeze-dried soluble coffee has a longer shelf life than spray-dried soluble coffee used in this study but the reason for this difference was not clear. It was established (Sivetz, 1963) that moisture content of soluble coffee is a critical factor for keeping quality. He stated that the flavor of the product is relatively stable at 3% moisture content. Moreover, Lee (1965) reported that moisture in air on even a fairly dry day can hydrolyze some of the esters, acetals, and ketals in coffee aroma to less flavorsome constituents. It was found that the commercial spray-dried coffee used in this study had a moisture content of 4.5% while the freeze-dried coffee had only 2.0% moisture. The tentative conclusion we draw is that moisture content appears to be more critical to the keeping quality of soluble coffee than headspace gas treatment. This is in agreement with a statement by Sivetz and Foote (1963). For spray-dried soluble coffee it seems prudent, that when the product moisture content exceeds a certain level, it should be packed under lower oxygen conditions. The picture of what happens to soluble coffee of different moisture contents and stored in different atmospheres is based on deductions from published results. The subject of these inter-relationships will require more extensive research.

Table 3–Consumer panel testing of soluble coffees served black–hedonic rating[a]

Headspace treatment	Initial	12 months 37.8°C	
	Spray-dried soluble		
Air	5.7[b]	4.9*	
2% Oxygen	5.7[b]	5.8	NS[c]
0% Oxygen	5.7[b]	5.3	
Control (new sample)	NA[d]	5.4	
	Freeze-dried soluble		
Air	6.0[b]	5.8	
2% Oxygen	6.0[b]	5.3	NS[c]
0% Oxygen	6.0[b]	5.8	
Control (new sample)	NA[d]	5.7	

[a] 5 = neither like nor dislike; 6 = like slightly
[b] Single determination
[c] NS = not significant
[d] NA = not applicable
* Significant difference at 5% level; LSD = 0.6

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