RETENTION OF VOLATILES IN FOAM-MAT DRIED AND FREEZE-DRIED ORANGE JUICE

ROBERT E. BERRY AND JAMES L. FROSCHER[1]

U. S. Fruit and Vegetable Products Laboratory[2]
Winter Haven

ABSTRACT

Cold pressed peel oil was added to high quality 50°Brix commercial orange concentrate and foam-mat dried, or diluted to single strength juice and freeze-dried using different platen temperatures. Amounts of peel oil ranged from 0.026% to 0.106% for samples to be foam-mat dried, and from 0.011% to 0.109% for freeze drying. For foam-mat drying, previously developed standard commercial conditions were used and for freeze-drying, samples were prepared using different platen temperatures. Among the foam-mat dried samples, that to which the lowest oil had been added yielded 62% retention in the dried product. The sample to which 0.054% oil had been added yielded 56% retention and the sample to which 0.106% oil had been added retained 73% in the dried product.

Among the freeze-dried samples, proportions of retained oil were higher in powder prepared from concentrations with the highest initial oil levels. With a given initial oil level, although variable, there was no consistent difference in oil retention between samples dried at different platen temperatures so long as they did not melt. In the freeze-dried samples, COD of volatiles ranged from 19 to 28% of original values, while only 7% were retained in foam-mat dried samples.

INTRODUCTION

The retention of volatiles in food during dehydration processes is becoming of increasing interest as the manufacture of dehydrated foods becomes more widespread. Recent studies on volatility of flavor compounds during freeze-drying and vacuum drum drying have been carried out mainly on model systems (Saravacos and Moyer, 1966, 1968). They found that 50 to 75% of added esters could be retained in a freeze-dried gel model system if samples did not melt and surface temperature was kept below 50°C. Previous studies with foam-mat dried grapefruit juice have indicated 60% of added peel oil was retained during preparation of instant grapefruit juice on a crater-type foam-mat drier (Berry, et al. 1966).

Due to recent interests in the use of freeze dried orange juice for flavor enhancement (Berry and Froscher, 1968) and due to recent trends towards commercialization of foam-mat dried instant orange juice (IOJ), considerable need has developed for a study of the relative retention of volatiles in orange juice dried by these two methods. The development of a rapid and reliable method for testing for d-limonene (Scott and Veldhuis, 1966) suggested the use of this compound as an index of retention of volatile components. Although it contributes little to orange juice flavor, its boiling point places it in a range of volatility which is near the middle among the principle oil and essence components comprising a major part of orange juice flavor. D-limonene itself accounts for about 95% by volume of cold-pressed orange oil.

A study was made of orange oil and of water soluble volatiles added to FCOJ or single strength orange juice, and amounts retained in IOJ prepared by foam-mat drying using recommended commercial conditions, and by freeze-drying using different platen temperatures. This is a report of that study. The results of such work would indicate the relative advantages to be gained in using freeze-dried IOJ as a flavor additive, and in preparing freeze-dried IOJ under specific conditions.

MATERIALS AND METHODS

Preparation of IOJ

Foam-mat Drying.—The foam-mat dried samples of instant orange juice were prepared on a crater type drier as described by Berry, et al. (1965). Maximum product temperature was 160°F. Commercial cold-pressed peel oil was added to 50°Brix frozen orange concentrate (evaporator pump-out) until recoverable oil by

---

[1]Present address: S-M Vacu-Freeze Corp., Bartow, Fla.
[2]One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.
References to specific commercial products do not constitute endorsement.

analysis reached the desired level. Samples were prepared containing 0.026, 0.054, and 0.106% peel oil (ml oil/100 ml juice). The IOJ prepared from these samples was analyzed for moisture content and recoverable oil.

Freeze-drying.—Samples were prepared for freeze-drying by adding cold-pressed peel oil to 12.5°Brix orange juice which had been reconstituted from commercial FCOJ containing 10% fresh cut-back juice. In some experiments samples of fresh-frozen commercial single strength juice were used. Aliquots of oil were added until analysis indicated recoverable oil at the desired level. Oil levels tested were 0.011, 0.032, 0.041, 0.046, 0.083 and 0.109. Samples were frozen in trays with 2 sq. ft. surface, approximately ½-in. deep. Samples were frozen in a −90°F freezer, then placed in a pilot model shelf freeze-drier, Thermovac Freeze Dryer Model 10-DR (with balance), Thermovac Industries Corporation, Copiague, New York. Samples were dried with controlled platen temperatures of 70, 80, 90, 100 and 120°F, respectively. All samples were dried for seven hours and the final products were analyzed for moisture content, recoverable peel oil as d-limonene, and chemical oxidation demand (COD).

Analytical Methods

Oil analysis.—Samples were analyzed for recoverable oil by the Bromate Titration Method (Scott and Veldhuis, 1966).

Moisture analysis.—Foam-mat and freeze-dried samples of IOJ were analyzed for moisture by a modification of the Karl Fisher titration method (McComb and Wright, 1954).

Chemical oxidation demand.—Samples of FCOJ and freeze-dried IOJ were analyzed for chemical oxidation demand of water soluble volatile constituents by the chromate oxidation method of Dougherty (1968). In this method the juice reconstituted from FCOJ or IOJ was distilled with the aid of an oil trap. The oil-free distillate containing water-soluble volatiles was oxidized by potassium dichromate-H_2SO_4. The chemical oxidation demand was determined in ppm by measure of optical density and reference to a standard curve.

Results and Discussion

The retention of oil in foam-mat dried IOJ is given in Table 1. The sample to which 0.026% oil had been added yielded 62% retention and resulted in a powder containing 1.9% moisture. The sample with about double this level of oil resulted in 56% retention and 1.7% moisture content. The sample with 0.106% oil resulted in a higher percentage retention, 73% and a final moisture content of 1.6%. The two lower oil level samples required no special treatment for foam-mat drying. The highest oil level was somewhat difficult to foam satisfactorily, however, and required an increase in foaming agent (methylcellulose 10 cps, Dow Chemical Company, Midland, Michigan) from 0.45% used in the other samples to 0.75%. The fact that moisture was more difficult to remove from these samples is indicated by the increase in moisture content of the final product. The control sample, with no added oil had a moisture content of 1.2%. It is interesting to note that in all samples, more than half the oil added to the concentrate was retained in the final product.

TABLE 1. RETENTION OF OIL IN FOAM-MAT DRIED IOJ

Oil Level %		Retention	Moisture
Original	Final	%	%
0.026	0.016	62	1.9
0.054	0.030	56	1.7
0.106	0.078	73	1.6

The results of the oil retained in freeze-dried IOJ are given in Table 2. No general trends of oil retention were observed which would appear related to platen temperature. Highest oil levels

TABLE 2. RETENTION OF OIL IN FREEZE-DRIED IOJ.

Platen Temp. °F.	Oil Level %		Retention	Moisture
	Original	Final	%	%
120	0.011	0.003	26	4.3
	0.041	0.012	30	3.9
	0.083	0.041	49	3.6
110	0.011	0.006	48	5.0
	0.041	0.012	30	4.3
	0.083	0.039	47	4.8
100	0.011	0.007	54	5.5
	0.032	0.013	31	5.1
	0.046	0.012	26	5.1
	0.083	0.041	49	5.2
90	0.011	0.004	26	6.4
	0.032	0.011	34	6.6
	0.046	0.017	42	5.7
	0.109	0.043	40	6.9
80	0.013	0.004	29	4.4
	0.032	0.010	33	6.1
	0.046	0.014	30	5.0
	0.109	*	*	*
70	0.013	0.005	44	4.9
	0.032	0.006	18	6.5
	0.109	*	*	*

*Highest oil samples could not be dried at these lower platen temperatures due to melting.

generally resulted in approximately 45 to 50% retention, middle *oil* levels in approximately 25 to 35% retention, and lowest oil levels from 25 to 45% retention. There was considerable variation in percent retention, and widest variation was among the lowest oil level samples. In these samples smaller absolute variations amount to larger percentages. As expected moisture content of the final product was generally higher as the platen temperature was lowered. It is interesting to note that only in one sample, 0.011% original oil level dried at platen temperature 100°F, was the final oil retention above 50%. It is also interesting to note that the highest oil level, 0.109% could not be dried at the lower platen temperatures, 80°F, and 70°F due to melting. Thus, oil content was not as completely retained in freeze-dried IOJ as in foam-mat dried IOJ even though foam-mat dried samples were more completely dried.

The retention of water soluble volatiles as measured by chemical oxidation demand (COD) from foam-mat and freeze-dried samples is shown in Table 3. Almost all water soluble volatiles were lost in the foam-mat dried samples as indicated by the reduction from 585 ppm COD in the original essence fortified concentrate to 42 in the IOJ. For the freeze-dried samples, however, which consisted of commercial fresh-frozen cut-back SSOJ, the retention of water soluble volatiles ranged from 19 to 28%. The highest retention was obtained in samples dried at a platen temperature of 110°F., with about 28%. Retention at lower platen temperatures ranged from 10 to 24%. The lowest percent retention (19%) of water soluble volatiles from freeze-dried IOJ was produced at a platen temperature of 90°F.

These results indicate there was a higher retention of oil-soluble volatiles, represented by d-limonene, during foam-mat drying than during freeze-drying. However, with regard to water soluble volatiles, there was a reasonable retention during freeze-drying but virtually complete loss during foam-mat drying. If a considerable degree of fresh juice flavor is contributed by the water-soluble volatiles, then these observations may help explain the reasons for improved fresh-juice flavor which has been observed in freeze-dried IOJ (Berry and Froscher, 1968).

TABLE 3. RETENTION OF WATER SOLUBLE VOLATILES FROM FOAM-MAT AND FREEZE-DRIED IOJ AS MEASURED BY CHEMICAL OXIDATION DEMAND (COD)

Process	Temp. °F	COD (ppm) Original	Final	Retention %
Foam-mat	160	585	42	7
Freeze-dried	120	563	123	22
	110	563	155	28
	100	563	133	24
	90	563	107	19
	80	563	133	24
	70	563	133	24

This study, therefore, indicates freeze-drying as the preferred method where retention of water soluble volatiles is of primary concern, and foam-mat drying where oil soluble volatiles are critical. In systems such as orange juice, where both types are important to flavor, the use of freeze-dried aroma reinforced flavor adjuncts to foam-mat IOJ may represent the optimum usage of both drying methods.

LITERATURE CITED

1. Berry, Robert E., O. W. Bissett and C. J. Wagner, Jr. 1966. Flavor fortification and storage of foam-mat dried grapefruit crystals. Proc. Fla. State Hort. Soc. 79: 330-4.
2. Berry, Robert E., and J. L. Froscher. 1968. Extent and rate of drying, particle size relationships and storage studies on freeze-dried citrus juices. Proc. Fla. State Hort. Soc. 81: 254-8.
3. Berry, Robert E., O. W. Bissett, C. J. Wagner, Jr. and M. K. Veldhuis. 1965. Faom-mat dried grapefruit juice. Time-temperature drying studies. Food Technol. 19(3): 126-8.
4. Dougherty, M. H. 1968. A method for measuring the water-soluble volatile constituents of citrus juices and products. Food Technol. 22(11): 1455-6.
5. McComb, E. A. and H. M. Wright. 1954. Application of formamide as an extraction solvent with Karl Fischer Reagent for the determination of moisture in some food products. Food Technol. 8: 73.
6. Saravacos, G. D. and J. C. Moyer. 1966. Volatility of some flavor compounds during freeze-drying of foods. N. Y. Ag. Experiment Station Journal Paper No. 1533, Presented at A. I. Ch. E., Detroit, Dec. 7.
7. Saravacos, G. D. and J. C. Moyer. 1968. Volatility of some aroma compounds during vacuum-drying of fruit juices. Food Technol. 22(5): 89-93.
8. Scott, W. Clifford and M. K. Veldhuis. 1966. Rapid estimation of recoverable oil in citrus juices by bromate titration. J.A.O.A.C. 49: 628-33.