

STORAGE STUDIES ON FROZEN CITRUS CONCENTRATES*

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The wide acceptance of orange juice concentrate, a new frozen food made by low temperature evaporation. (1), suggested the advisability of publishing observations on its storage characteristics. Briefly, the concentrate is made from fresh orange juice by boiling off water at about 55°F. in vacuum concentrators. The concentrate, having 50 percent by weight of solids, is diluted to 42 percent by addition of fresh juice. The material is then chilled to 15 to 20° F., filled into cans, frozen, and held at 0° to -10°F. The consumer partially thaws the material and reconstitutes by mixing three volumes of water with one of concentrate. Further details of the process have recently been reviewed by Burton (2). The concentrate should not be confused with the conventional product made in typical vacuum pans at a temperature of 100° F. or higher.

As pointed out previously (2, 4) use of fresh juice to provide better flavor and aroma in frozen concentrates was developed by MacDowell, Moore, and Atkins of the Florida Citrus Commission, working at the U. S. Citrus Products Station in Winter Haven, Florida. A comprehensive review

of the literature and commercial practice in this field up to 1945 is given by Moore, Atkins, Wiederhold, MacDowell, and Heid (7).

The stability of citrus juice concentrates made at low temperatures is of special importance. The data to be presented indicate that the taste and ascorbic acid content retained closely approximate fresh raw juice rather than canned pasteurized single-strength juice. To maintain this quality, the product is kept in storage at approximately 0°F. However, consumers may hold the product at higher temperatures for varying periods of time. The present report, therefore, gives data for storage at several temperature levels. The effect of several processing variations is also discussed.

PROCEDURE

Batches of concentrate were made in a pilot plant evaporator at approximately 55°F. and packaged in tin cans with and without added fresh single-strength juice. For certain experiments, described more fully below, some of the concentrate was pasteurized. Unless otherwise indicated, the plain tin cans of five ounce capacity were sealed under 27 inches of vacuum and stored at 0°F. in a small walk-in cold room. Constant temperature boxes were constructed for other storage temperatures. Occasionally, commercially prepared concentrate was studied. This was made also by low temperature evaporation (2). This product requires a far shorter processing time than that used in the pilot plant, and the taste was definitely far superior to the concentrate made in the pilot plant. Storage studies were scheduled for six months or one year.

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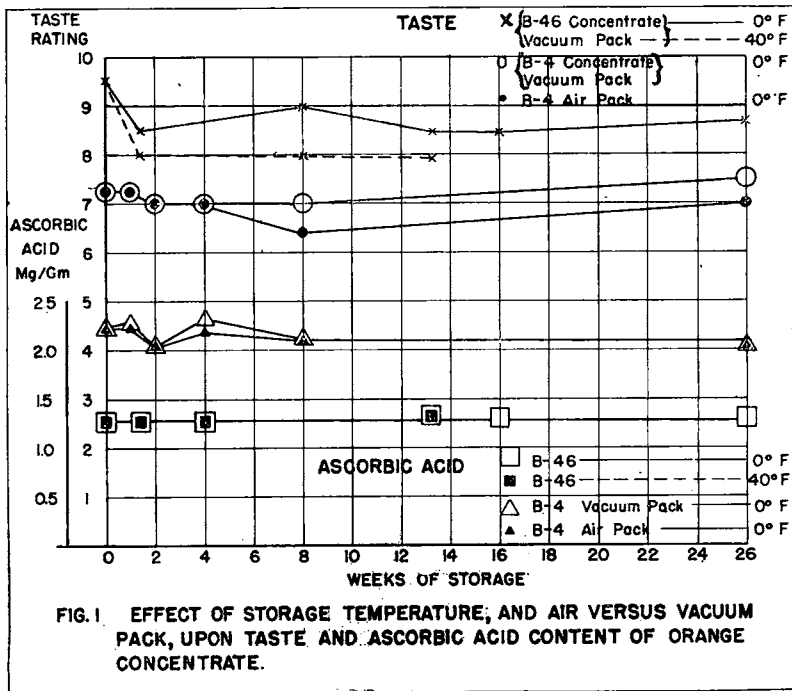
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Taste ratings were made on all samples using a range from 1 to 10; where 10 represents an excellent raw freshly-extracted juice; 8, a reconstituted juice of good quality; 7, a juice of fair quality when compared to raw fresh juice; 6, a minimum level of acceptability for a commercial concentrate; 4, a poor product; and 0 to 2, a

case of pasteurized products or products stored for long periods at high temperatures, several different off-tastes appeared; thus, one taster sensitive to a very slight fermented odor would show a marked preference for a pasteurized product while those sensitive to a "processed" taste reacted in just the opposite direction. In general, the members

Fig. 1



foul tasting one. All samples were evaluated for aroma, but since the values were almost identical to taste ratings, they are not given. Three or more people independently evaluated the samples for taste and aroma; the values were averaged for use in the tables and graphs. Where sharp differences among tasters arose, however, the separate evaluations were retained. In the

of the taste panel checked each other within one-half taste unit. For any given experiment, the samples were tasted side by side, in coded beakers so that the tasters did not know what the samples were, and the emphasis was upon differences between samples. As to the absolute magnitude of the taste ratings, this varied because of lack of taste memory. In a laboratory where

for months several storage experiments on orange concentrate and orange powder from different batches of oranges were started each week, one found it hard to be sure whether a given sample should be called 8 or 7.5. Differences between samples were easy to detect, however, which served the purpose of the experiments. The tasters

with periodic checks by the modifications of Robinson and Stotz (10) and Rubin, Jahns, and Bauernfeind (11) for reductions, dehydroascorbic acid, and metal interferences. Daily standardizations were made with crystalline ascorbic acid. Negligible amounts of dehydroascorbic acid were found and thus, unless otherwise noted,

Fig. 2

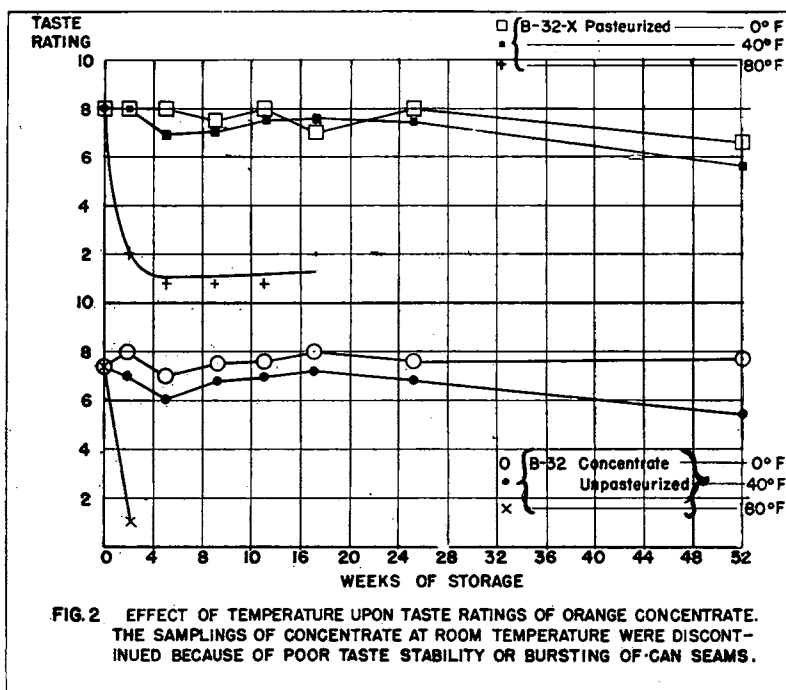


FIG. 2. EFFECT OF TEMPERATURE UPON TASTE RATINGS OF ORANGE CONCENTRATE. THE SAMPLINGS OF CONCENTRATE AT ROOM TEMPERATURE WERE DISCONTINUED BECAUSE OF POOR TASTE STABILITY OR BURSTING OF CAN SEAMS.

were periodically checked for their reproducibility. Only men who could accurately reproduce their evaluations were retained on the panel.

Citric acid and pH were determined; the latter data showed no notable changes and are not included except where differences were observed. Ascorbic acid was determined by the method of Bessey and King(1)

data in this report represent reduced ascorbic acid.

RESULTS

Temperature of storage is indeed important for an unpasteurized product such as a frozen food. Figure 1 gives a comparison between storage temperatures of 0° and 40° F. The taste ratings were generally 0.5 to 1.0 taste rating lower for concentrate held

at 40° F. than for concentrate held at 0° F. The differences in taste were more marked for a commercial product containing added fresh juice such as used in experiment B-46, which had an initial taste rating of 9.5, than they were in experiment B-18, Table 1, where a pilot plant concentrate made of early season juice was used.

mental Can Company for the analyses in the columns headed by the initials C. C. C. and an asterisk. Their taste data represent averages of six people in a taste panel. Usually, as was the case also in experiment B-46, the taste ratings dropped progressively in going from 0° to 20° to 40° to 75°.

Ascorbic acid analyses, presented in

Fig. 3

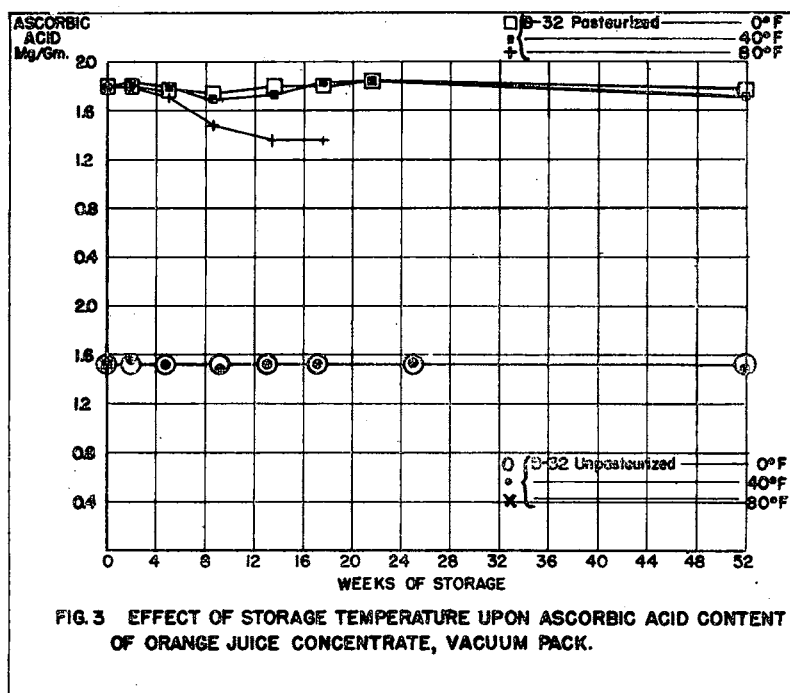


FIG. 3 EFFECT OF STORAGE TEMPERATURE UPON ASCORBIC ACID CONTENT OF ORANGE JUICE CONCENTRATE, VACUUM PACK.

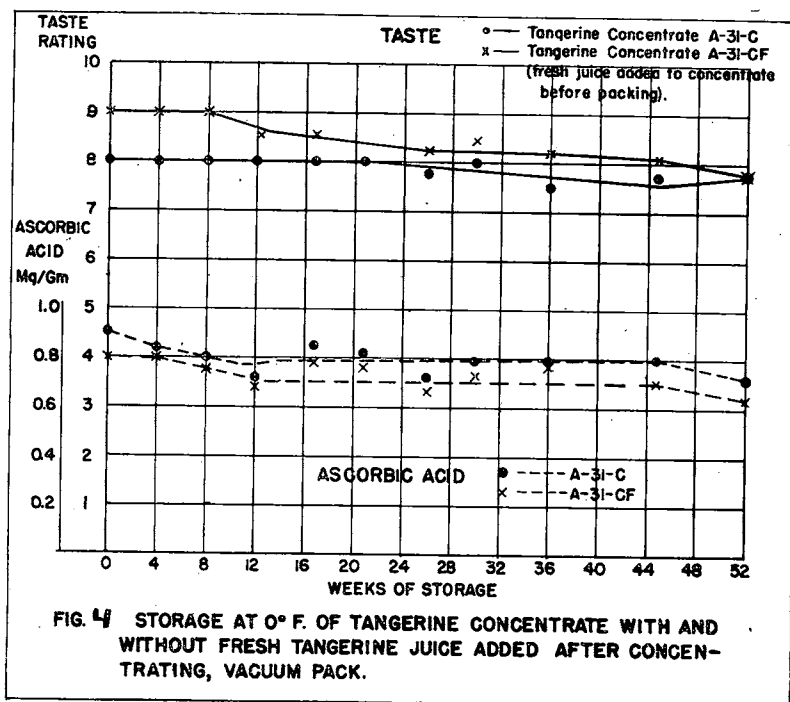
In experiment B-18, Table 1, fresh juice was not added back to the concentrate after concentration or before canning. Table 1 gives data for taste, ascorbic and citric acids for experiment B-18 which was packed cooperatively in Plymouth by this laboratory and John Boyd and Gordon Chissom of the Continental Can Company. We are indebted to W. J. Mutschler of the Conti-

Table 1 and Figure 1, indicate no loss, within experimental limits, in samples B-46 at 0° for six months. The B-46 (Figure 1) sample was stored only four months at 40° F. at which time no loss in ascorbic acid was observed. In experiment B-32, Figure 3, orange concentrate stored one year is seen to lose very little ascorbic acid. The initial figure was 1.53 milligrams per gram

of concentrate while after one year at 0°, B-32 had 1.50 mg/g as determined by the Bessey and King technic. A test for reductones (10)—materials other than ascorbic acid which reduce the indophenol dye used in titration—was negative while interferences caused by tin or iron (10) were found to be equivalent to 0.03 mg of ascorbic acid

ten or fifteen per cent higher than we report. Both laboratories used indophenol titration. An additional experiment, B-24, was stored at 0°, 10°, 20°, 30°, and 40°F. and is not presented in detail because of lack of space. After one year, the losses ranged from one per cent at 0° F. to three per cent at 40° F. Moore *et al* (8) have reported

Fig. 4



per gram. Thus, the drop for one year was from 1.53 to 1.47, or less than four per cent of the total amount present. The values for B-32 at 40° were comparable as were those for B-32-X, pasteurized, at both temperatures. Somewhat greater differences were found in one experiment, B-18. It will be observed that the data for ascorbic acid analyses reported by Mutschler (9) were

that commercially packed single-strength orange juice in tin cans shows an ascorbic acid loss of about 5% when stored at 40° F. for six months. However, they report an 18% loss when the juice is stored six months at 80° F., the standard commercial storage temperature.

Figure 2 shows changes in taste as a function of time and temperature of storage. The

taste, ratings were most stable at 0°F., slightly less at 40°F., while at 80°F. the concentrate was soon unpalatable. After one year the pasteurized product had a rating of about one taste unit lower than the unpasteurized product.

Figures 4 and 5 show ascorbic acid retentions in concentrates made from tange-

the advantage of addition of fresh juice back to concentrate. In this case, concentrates were made having 50 per cent solids and fresh juice was added to bring the solids to 42 per cent. The taste was improved by one taste unit for tangerine juice and 0.5 units for the grapefruit-orange blend. The difference between the tastes

Fig. 5

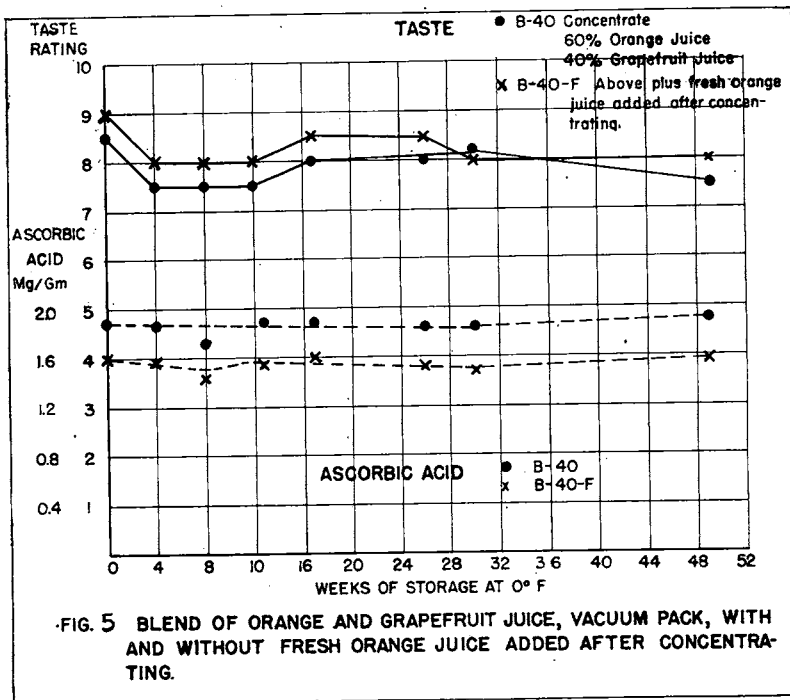


FIG. 5 BLEND OF ORANGE AND GRAPEFRUIT JUICE, VACUUM PACK, WITH AND WITHOUT FRESH ORANGE JUICE ADDED AFTER CONCENTRATING.

rine juice and from a blend of orange and grapefruit juice. The stability of ascorbic acid in tangerine juice is evidently less than that in orange juice or in a mixture of orange juice and grapefruit juice. Not shown for lack of space are data on lime juice concentrate which were similar to those found for tangerine concentrate.

Figures 4 and 5 graphically demonstrate

tends to be less as storage proceeds, suggesting that the volatiles restored by addition of fresh juice are somewhat less stable than the other less volatile flavor constituents.

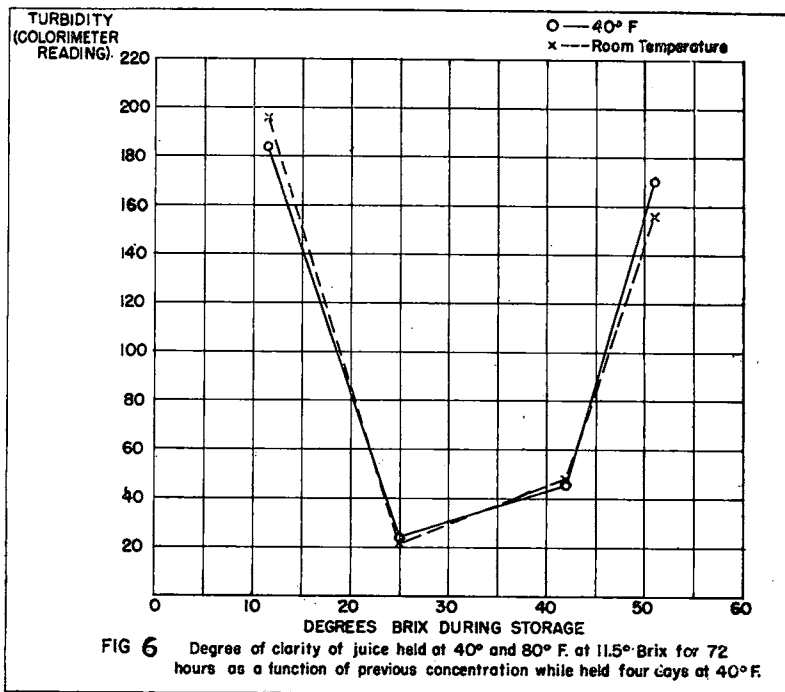
Table 1, Experiment B-18, shows variations in citric acid during storage. The experiment compares (B-18-A) hot-filled concentrate pasteurized 3.3 seconds at 85°F.

with cold-filled unpasteurized concentrates, (B-18-D) nitrogen-packed, (B-18-E) vacuum packed, and (B-18-F) air-packed. During storage, there is a trend toward higher acid values in alphabetical order of the treatments; (A) through (F). No regular variation was observed with changes in temperature, but in all samples there was

in citric acid in the remaining experiments were small and are not shown for lack of space.

Clarification of citrus juices is an important commercial factor. One often observes that freshly extracted orange juice becomes unsightly in appearance after standing several hours at room temperature. Gross

Fig. 6



a slight decrease in acidity. Since the standard sodium hydroxide was periodically standardized with potassium acid phthalate and found to be stable, the slight differences which occur were felt to be true differences. It was felt that possibly the greater amount of oxygen in the air-packed samples might be associated with oxidation of some organic compound to a titratable acid. Changes

particles of pulp settle out and the colloidal yellow "cloud" becomes a clear light yellow serum. Joslyn and Sedky (5) have reviewed the problem and showed that the enzyme associated with clarification can be partially inhibited or almost completely inactivated by heating to 80°C. for several minutes. In this laboratory, juice pasteurized several seconds at or above 205°F., the common

TABLE 1.
STORAGE OF ORANGE JUICE CONCENTRATE
Weeks in Storage, Experiment B—17

Sample	No.	Initial	Initial C.C.C.*	2.5 Weeks			4 Weeks			4 Weeks C.C.C.*		8 Weeks		16 Weeks		16 Wks C.C.*	
				0°	20°	40°	0°	20°	40°	0°	40°	0°	20°	0°	40°	0°	40°
Taste Rating	A	7+	5.5	7.5	7.5	7.5	7	6.5	6.5	5.7	5.7	8	7	8	6.4	6.3	
	D	7+	7.7	6.5	—	7.5	7.5	—	7	7.8	—	8	—	7.5	7.9	—	
	E	7+	8.1	7.0	7.5	7	7	7	6.5	5.7	7.9	7	7.5	7	7.9	7.8	
	F	7+	7.6	7.5	7.5	7.5	7	7	7	7.4	7.7	8	7.5	7.5	8.0	7.4	
Ascorbic Acid mg/g	A	2.14		1.78	1.75	1.78	1.86	1.84	1.82			1.76	1.83	1.74			
	D	2.14		1.78	—	1.81	1.82	—	1.81			1.68	—	1.75			
	E	2.14		1.88	1.77	1.78	1.83	1.99	1.83			1.80	1.92	1.79			
	F	2.14		1.92	1.72	1.83	1.80	1.86	1.90			1.77	1.87	1.80			
Percent Citric Acid	A	4.21		3.78	3.85	3.81	3.86	3.89	3.82			3.83	3.79	4.00			
	D	4.21		3.79	—	3.88	3.94	—	3.94			3.74	—	4.05			
	E	4.21		4.09	3.95	3.79	4.00	4.27	3.90			3.98	4.16	4.27			
	F	4.21		4.10	3.71	3.86	3.85	3.95	4.09			3.97	4.06	4.27			

*Data from Continental Can Company.

¹Sample A was concentrate heated 3.3 seconds at 185° F., filled hot in 5 ounce cans and dropped into ice water two minutes after filling.

Samples D, E and F were not pasteurized and were filled cold.

Sample D was placed in a vacuum chamber brought to 28.5 inches of vacuum. Nitrogen was introduced and the cans were sealed at approximately atmospheric pressure.

Sample E was vacuum packed at 28.5 inches vacuum.

Sample F was sealed with air in the headspace of the can.

TABLE 1—Continued

Sample	No.	26 Weeks			26 Weeks C.C.C.*			38 Weeks	52 Weeks	52 Weeks C.C.C.*		
		0°	20°	40°	0°	40°	75°	0°	0°	0°	40°	75°
Taste Rating	A	7.5	7.5	7.5	6.1	6.6	2.8	7	7	7	6.8	4
	D	8	—	7+	—	—	—	8	8+	—	—	—
	E	8	7.5	7	7.4	7.7	—	8	8+	8.0	7.4	—
	F	8	7.5+	7	7.5	6.7	—	8-	8	7.6	6.9	—
Ascorbic Acid mg/g	A	1.70	1.76	1.75	—	—	—	1.73	1.70	2.01	2.06	0.85
	D	1.75	—	1.76	—	—	—	1.71	1.70	—	—	—
	E	1.82	1.84	1.79	—	—	—	1.78	1.81	2.02	2.15	—
	F	1.85	1.81	1.86	—	—	—	1.82	1.78	2.09	2.03	—
Percent Citric Acid	A	3.84	3.88	3.79	—	—	—	3.67	3.68	—	—	—
	D	3.94	—	3.95	—	—	—	3.78	3.60	—	—	—
	E	4.10	4.16	3.89	—	—	—	3.90	3.96	—	—	—
	F	4.14	3.95	4.13	—	—	—	4.00	3.88	—	—	—

*Data from Continental Can Company

practice in Florida with concentrate for export, showed no tendency to clarify. Thus, pasteurized juice clarifies slowly or not at all.

It would be expected that the concentrates described here would be subject to extremely rapid clarification. Qualitatively, we have observed fresh juice to clear in much shorter time than the concentrates made from it. Specifically, samples of fresh Valencia orange juice and the concentrate made from it were brought up to 86°F. and stored at room temperature with added sodium benzoate. The concentrate was taken from the can line just before being sealed. It was reconstituted to the original fresh juice concentration. After six hours, the fresh juice had a line separating a turbid cloud above from a dark orange, more opaque base below. The reconstituted juice showed no separation. After 22 hours, the fresh juice had settled further and the "cloud" above had noticeably cleared while the reconstituted concentrate still gave no evidence of clearing.

Clarification rates in orange juice after reconstitution to 11.5° Brix, were studied as a function of concentration of orange juice solids during storage at 40°F. An orange concentrate, 15 months old, was removed from the 0°F. cold room. It was stored at various concentrations for four days at 40° F. The concentrations studied were 51° Brix, its original strength, and 42, 25, and 11.5° Brix made up with sterile water and placed in sterile tubes at (a) 40°F. and (b) 80°F., room temperature. After three days, the turbidities were read in a colorimeter as per Loeffler (6). The higher the reading the more turbid was the juice. Figure 6 shows that clarification was most rapid in the samples stored at 25° Brix, next most at 42° Brix. Unexpected was the fact that previous storage at 11.5 and 51° Brix at 40°F, was associated with very slight clarification. This was observed for the same concentrations in a previous experiment with fresh concentrate. Cruess et al (3) reported that

adding sugar to lemon juice retarded clarification. In both experiments, cans were stored both full and one-half full. In the latter, there was a tend toward very slightly lower rates of clarification. Figure 6 indicates, finally, that the rates of clarification of the reconstituted juice were almost identical at 40°F. and room temperature. Although the above was carried out on two different samples of concentrate with identical results, it would be of interest to repeat clarification tests in another season on several varieties of oranges.

A series of seven samples representing commercial operation during 1947 up to mid-April were tested for rate of clarification of reconstituted juice, Table 3. These samples represented concentrate made from various early-, mid-, and late-season juices which had been stored at 0° to -10°F. up until the time of testing. A total of seven samples of frozen concentrate were removed from the cold room and thawed as rapidly as possible in warm water. A portion of each of the seven samples was then reconstituted and kept at refrigerator temperature (51°F.) in test tubes for observation over a 24-hour period. The unreconstituted remainder of each thawed concentrate sample was kept in the refrigerator (51°F.). At the end of 24 hours, 48 hours, and 72 hours, portions of each sample of thawed concentrate were reconstituted and observed.

All samples tested held up for 24 hours after reconstitution when such reconstitution was done immediately after thawing. When reconstituted after having thawed 24, 48, and 72 hours at 51°F., behavior of the reconstituted product became progressively worse. The sample made up after 72 hours of thawing separated almost immediately upon reconstitution and was the only sample observed that gave a completely clear upper liquid level.

In short, 0°F. (or lower) was the best storage temperature for concentrate to protect flavor and physical stability after reconstitution.

Vacuum-packed concentrate was compared to air-packed in experiment B-4, Figure 1, and experiment B-17, Table I. After 8 weeks at 0°F., the orange concentrate packed under vacuum was superior to the air-packed product, Figure 1. The differences were not consistent until after 26 weeks in experiment B-18. Thus, at 38 and 52 weeks, concentrates packed in nitrogen or under vacuum were superior in taste to those sealed with air in the headspace. The magnitude of the differences among the various packing methods was approximately one-half taste unit for these two batches of concentrate, neither of which had fresh juice added back. In view of the behavior of tangerine juice, where after a year, the advantage of added fresh juice was lost, a short experiment with commercial concentrate containing added fresh juice was carried out. A uniform batch of concentrate was (a) air-packed, (b) packed under 27 inches vacuum, and (c) packed with nitrogen after evacuating to approximately 29 inches of vacuum. The initial taste rating was 9.5. After one month at 0°F., the taste ratings were 9, 9.5, and 9 in the above order while after five months, they were 8+, 9, and 8.5+, respectively. Perhaps the nitrogen pack suffered from loss of volatiles owing to the higher vacuum employed. There was some frothing in the cans during evacuation. The advantage of the vacuum pack, however, was decisive.

CONCLUSIONS

Commercially prepared frozen orange juice concentrate exhibits excellent retention of aroma, taste, and ascorbic and citric acids during storage at 0°F. Concentrates stored at temperatures as high as 40°F. retain ascorbic acid almost as well as at 0°F., but flavor is degraded somewhat. Negligible amounts of dehydro-ascorbic acid are present in orange concentrate stored for long periods of time. Storage at 40°F. is a distinct disadvantage if the reconstituted

juice is not to be consumed immediately since enzyme action results in clarification and separation of the colloidal constituents producing an unsightly appearance. The low-temperature fast-cycle concentrating process reduces enzyme action slightly. Samples of orange concentrate stored a year at 0°F. have a stable "cloud" when reconstituted.

Air had a detrimental effect upon the storage stability of orange concentrate. Flavor retention is better in vacuum-packed cans than in air-packed cans.

REFERENCES

1. BESSEY, O. A., AND KING, C. G. The distribution of vitamin C in plant and animal tissues, and its determination. *J. Biol. Chem.* 103. 687-698 (1933).
2. BURTON, L. V. High vacuum technics utilized for drying orange juice. *Food Industries* 19. 617-622, 739-743 (1947).
3. CRUESS, W. V., SEAGRAVE-SMITH, H., AND GLAZEWSKI, I. G. A. Frozen concentrates makes fresh lemonade. *Quick Frozen Foods* 99, 86-87 (1947).
4. HAYES, N. V., COTTON, R. H., AND ROY, W. R. Problems in the dehydration of orange juice. *Proc. A. Soc. Hort. Sci.* 47, 123-129 (1946); also *Food Packer* October, 1946.
5. JOSLYN, M. A., AND SEDKY, A. Effect of heating on the clearing of citrus juices. *Food Research* 5, 223-232 (1940).
6. LOEFFLER, H. J. Processing orange juice. *Ind. Eng. Chem.* 33, 1308-1314 (1941).
7. MOORE, E. L., ATKINS, C. D., WIEDERHOLD, E., MACDOWELL, L. G., AND HEID, J. L. The concentrating and drying of citrus juices. 1945 *Proc. Inst. Food Technologists* 160-168 (1946).
8. MOORE, E. L., WIEDERHOLD, E., AND ATKINS, C. D. Changes occurring in orange and grapefruit juices during commercial processing and subsequent storage of the glass- and tin-packed products. *Fruit Products J.* 23, 270-175, 285. (1944).

9. MUTSCHLER, W. J., Personal communications. *Continental Can Company, April, 1947.*
10. ROBINSON, W. B., AND STOTZ, E. The indophenol-xylene extraction method for ascorbic acid and modifications for inter-
ing substances. *J. Biol. Chem.* 160, 217-225 (1945).
11. RUBIN, S. J., JAHNS, F. W., AND BAUERNFEIND, J. C. Determination of vitamin C in fruit products. *Fruit Products J.* 24, 327-330, 344, 350 (1945)

REPORT ON CITRUS BEVERAGE BASE RESEARCH

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SUMMARY

This report reviews studies by Research Fellows of the Florida Citrus Commission on the production of bases for blended citrus juice beverages. Orange, grapefruit, and lime concentrates were investigated in preparing a variety of "ade" bases designed to yield a beverage containing a minimum of 30 percent of juice. The sugar, acid and essential oil components are reviewed; emulsifying and dispersing agents are discussed.

INTRODUCTION

The consumption of non-alcoholic bottled beverages in this country has created a multi-million dollar industry. Estimates furnished by the American Bottlers of Carbonated Beverages, Washington, D. C. (1) indicates a retail consumption increasing from \$760,000 in 1849 to \$589,849.56 in 1946. In 1849 36,000,000 bottles of non-alcoholic beverages were consumed by the American public. In 1946 this figure reached the staggering total of 17,695,000,000. The per capita consumption of bottled soft drinks has grown from 1.6 in 1849 to 125.3 in 1946.

In recent years beverages containing

fruit juice have contributed to this volume. The use of fruit juices and concentrates in beverages was reported by Cruess and Irish in 1923 (2), and by Irish in 1925 (3). Cruess and Aref (4), and Bailey (5) reported on the composition of fruit juice beverages during the period 1933-1936. These beverage flavors included apple, lemon, grapefruit, orange, grape, pineapple, cherry, loganberry, strawberry, and raspberry. However, of the fruit juice beverages that have been introduced, only citrus juice beverages, orange, lemon, lime and grapefruit became widely popular. Bailey (5) in reporting the composition of orange products, noted that the juice content (as estimated from the ash analysis) averaged 15 percent. Later analysis by Bailey (6) (7) indicate juice contents for orangeade beverages of 12-20 percent (1939) and 14-32 percent (1940).

On June 4, 1947, the Florida Citrus Commission authorized research on citrus beverage bases, stipulating that initial studies should be toward the formulation of a citrus "ade" of (1) high juice content and (2) a character distinctive to Florida. It was also considered desirable to incorporate a maximum amount of grapefruit juice to assist in extending the market for this fruit.

Facilities for the project were furnished in cooperation with the University of Florida at the Citrus Experiment Station, Lake Alfred.

MATERIALS

The concentrated orange juice used in

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