proportions up to 50% of the total sugar used. When more than 50% of dextrose was used the dextrose crystalized out on freezing and gave a "bloom" to the puree that looked like mold.

Conclusions

From the data presented here it is concluded that, under the conditions of these tests, heat sterilized canned tangerine puree does not hold its flavor long enough to be practical as a flavoring material for ices, sherbets and ice cream. It is further concluded that, under the conditions of these tests, frozen tangerine puree holds its flavor without appreciable loss for at least two years and makes an excellent flavoring material for ices, puree, ice cream, cake filling and fountain drinks.

From the fact that, during consumer acceptance tests, the sale of frozen tangerine puree approximately double each year for three years, without advertising or promotion, at each of three outlets, it is concluded that frozen tangerine puree is a product that has met with good consumer acceptance.

Acknowledgdment

During the time that some of these tests were being run Mr. Owen W. Bissett, at the USDA Citrus Products Laboratory in Winter Haven, was also working on frozen tangerine puree, along with other citrus purees. Neither of us knew of the work being done by the other. Mr. Bissett called at my laboratory one day and mentioned the work that he was doing on frozen citrus purees. I told him of my work and was glad to adopt several helpful suggestions made by Mr. Bissett.

CLARIFICATION IN HEAT - TREATED PINEAPPLE ORANGE CONCENTRATES[1]

C. D. Atkins[2], R. L. Huggart[2] and F. W. Wenzel[3]

Florida Citrus Experiment Station

Lake Alfred

The stabilization of orange concentrate by heat-treatment during processing prior to freezing has been a subject of considerable study during the past several years in both commercial plants and research laboratories. The two principal reasons for heat treatment, either of the raw juice prior to concentration or of the concentrate during or after evaporation, are (a) the partial or complete inactivation of pectinesterase, the enzyme which may cause clarification and gelation, and (b) the destruction of acid-tolerant microorganisms that may cause spoilage during processing. In the commercial production of hot-pack concentrate for storage at refrigerated temperatures of 32° to 45°F., the practice has been to use sufficient heat treatment to provide complete enzyme inactivation and a commercially sterile product.

This report covers the results obtained from two investigations during the 1950-51 season on heat treatment of Pineapple orange juices and concentrates. The first study was concerned with the effect of the heat treatment of orange juice before concentration to 42°-Brix on pectinesterase activity, clarification and gelation during storage, and the destruction of microorganisms. The second investigation was undertaken to determine the temperature necessary for both the initial heat treatment of the orange juice and the final treatment of the concentrate in order that the hot-packed product could be stored at 32°F. without appreciable deterioration of quality. Because of the effect of heat treatment on the flavor of the product, it is desirable that the minimum amount of heat treatment necessary for obtaining the desired product stability be used.

Experimental Procedure

Heat Treatment of Juices prior to Concentration. — Pineapple oranges were thoroughly washed, the juice extracted in a Rotary press and finished in a Food Machinery (Model 35) finisher equipped with a 0.020 inch perforated screen. The pulp content of different batches of raw juices used was adjusted to

[1] Cooperative research by the Florida Citrus Commission and the Florida Citrus Experiment Station, aided by a grant from the American Can Company. Florida Agricultural Experiment Station Journal Series, No. 101.

[2] Florida Citrus Commission.

[3] Florida Citrus Experiment Station.

approximately 5, 10, and 15%. The juices were heated at temperatures from 125° to 195°F. by pumping through 40 feet of 3/16 inch I.D. stainless steel heating tube at a flow rate of one gallon per minute. Hot water was circulated around the heating tube. The time required to heat the juice from room temperature to the desired temperature was three seconds. The juice was discharged from the heater directly into a small pilot plant evaporator (1) thereby immediately lowering the temperature of the juice to 70°F. After concentration of the juice to 42°Brix, the concentrate was filled into six-ounce cans, frozen, and stored at —8°F.

Heat Treatment for Hot-Packed Concentrates. – Pineapple orange juice was extracted, finished and heat-treated at temperatures ranging from 160° to 195°F. in the same manner as described above. The pulp content of the raw juices was about 8%. After heating, the juice was immediately flashed into the evaporator and concentrated to 55°Brix. Each batch of the 42°Brix concentrate was prepared by the addition of heat-treated juice to the 55°Brix concentrate. Each of the cut-back juices was heated to the same temperature as that applied to the juice prior to evaporation. The heated cut-back juice was rapidly cooled to room temperature before blending with the 55°Brix concentrate. Prior to canning, the 42°Brix concentrates were individually heated in three seconds to either 165°, 175°, 185°, or 195°F., then filled directly into 9 1/2 ounce cans and closed in an American Can Company closing machine. The filled cans were water cooled immediately after closing to about 85°F. and then stored at 32°F.

Analytical Methods. – The degree of clarification in samples of the orange concentrates was determined by the method described by Huggart, Moore, and Wenzel (3) in which clarification is measured by determining the percentage light transmission of the centrifuged reconstituted juice. The method described by Olsen, Huggart, and Asbell (4) was used for determining the occurrence of gelation in the concentrates. A method quite similar to that described by Rouse and Atkins (5) was used to determine pectinesterate activity.

Experimental Results and Discussion

Heat Treatment of Orange Juice prior to Concentration. – Results reported in Table 1 show the effect of heat treatment of Pineapple ple orange juice before concentration to 42°-Brix on pectinesterase activity, clarification and gelation after storage at 80°F. for 24 hours, and destruction of microorganisms in the concentrate. Clarification and gelation

Table 1

Effect of Heat Treatment of Pineapple Orange Juice before Concentration to 42° Brix on Pectinesterase Activity, Clarification, Gelation, and Microorganism Count in the Concentrate [1]

Temperature °F.	Pectinesterase Activity % Inactivation, Pulp Content 5%	10%	15%	Degree of Clarification[2] After 24 hr. at 80°F., Pulp Content 5%	10%	15%	Degree of Gelation After 24 hr. at 80°F., Pulp Content 5%	10%	15%	Microorganism Count[3] X 1000, Pulp Content 5%	10%	15%
Unheated Control	—	—	—	Definite	Extreme	Extreme	None	None	Solid gel	208.0	24.0	47.0
125	—	—	—	Definite	Extreme	Extreme	None	None	Solid gel	27.0	28.5	40.0
135	—	—	—	Definite	Extreme	Extreme	None	None	Solid gel	225.5	24.5	26.0
145	33	12	4	None	Definite	Extreme	None	None	Semi-gel	13.0	18.0	85.0
155	50	32	34	None	None	Definite	None	None	Slight	11.0	10.5	8.0
165	75	70	78	None	None	None	None	None	None	2.6	3.5	2.2
175	87	96	96	None	None	None	None	None	None	4.9	1.9	1.1
185	100	100	98	None	None	Definite	None	None	None	9.2	7.5	5.7
195	100	100	100	None	None	None	None	None	None	5.3	6.0	3.2

1 The concentrates did not contain any cut-back juice.

2 Clarification measured by percentage light transmission of centrifuged reconstituted juice using Fisher Electrophotometer with Filter No. 650 and 23 ml. cylindrical cell. Degree of clarification: Less than 10% = None; 10-19% = Slight; 20-45% = Definite; 46-100% = Extreme.

3 Total count in reconstituted juice plated on dextrose agar, pH 7.

were prevented in these concentrates by heat treatment to 165°F. which brought about approximately 75% of pectinesterase inactivation and also resulted in a very marked decrease in the microorganism count. The effectiveness of heat treatment in preventing clarification and gelation was found to be dependent upon the temperature to which the juice was heated and the pulp content of the juice. The rate of clarification and gelation in the concentrates prepared from heat-treated juices was found to be primarily dependent upon the amount of pectinesterase still active in the juice, if inactivation had not been complete. Atkins, et al (2) recently discussed other investigations in which Valencia orange and Duncan grapefruit juices were heated to various temperatures prior to concentration. They stressed the importance of pH as a factor in the heat inactivation of pectinesterase and pointed out that a product could be stored at 40°F. for several months without clarification only if complete inactivation of the enzyme had been accomplished. In juices from different varieties of fruit and with different pH values, heat treatment prior to concentration to give complete inactivation was found to require temperatures in the range of 190°-210°F.

Clarification in Hot-Packed Concentrates.—Data presented in Table 2 show the stability to clarification of the hot-packed concentrates during storage at 32°F. over a period of 18 months. The pulp content of these products after processing was in the range of 4 to 5%, as determined by the centrifugal method, and the pH was 3.6. Pectinesterase determinations, made after six months storage at 32°F., showed only very slight traces of enzyme in these concentrates. Pectinesterase content in all of the 42°Brix concentrates ranged from none to 0.067 PE.units per milliliter. Gelation did not occur in any of the packs to a degree significant to consumers. The results shown in Table 2 indicate that final heat treatment of the 42°Brix concentrate at 185° or 195°F. was necessary to prevent clarification during storage at 32°F. for 12 months. Clarification did not occur, during 18 months at 32°F., in four of the 42°Brix concentrates which were finally heated at 195°F. This indicated that complete enzyme inactivation is necessary in heat-treated orange concentrates that are to be stored at 32°F. or higher for prolonged periods of time.

The effectiveness of initial and final heat treatments as a means of removing the last traces of enzyme activity is shown in Table 2. The initial heat treatment of both the raw

Table 2.

The Relation of Heat Treatment to Clarification of Hot-Packed Pineapple Orange Concentrate

Heat Treatment			Clarification during Storage at 32°F.				
Juice Prior to Concentration	Cut-back Juice	Concentrate 42°Brix	6 months	9 months	12 months	15 months	18 months
°F.	°F.	°F.					
160-165	160-165	165	Extreme	Extreme	Extreme	———	———
"	"	175	Extreme	Extreme	Extreme	———	———
"	"	185	None	None	Extreme	Extreme	Extreme
"	"	195	None	None	None	None	None
170-175	170-175	165	Extreme	Extreme	Extreme	———	———
"	"	175	Extreme	Extreme	Extreme	———	———
"	"	185	Extreme	Extreme	Extreme	———	———
"	"	195	None	None	None	None	None
180-185	180-185	165	Extreme	Extreme	Extreme	———	———
"	"	175	Extreme	Extreme	Extreme	———	———
"	"	185	Extreme	Extreme	Extreme	———	———
"	"	195	None	None	None	None	None
190-195	190-195	165	Extreme	Extreme	Extreme	———	———
"	"	175	Extreme	Extreme	Extreme	———	———
"	"	185	None	None	None	Extreme	Extreme
"	"	195	None	None	None	None	None

and cut-back juices in the range of 190°-195°F. did not prevent clarification, unless the 42°Brix concentrate was also heated to 195°F. The initial heat treatment at 160-165°F. was just as satisfactory provided that the final concentrate was heated to 195°F. Thus it would appear that initial heat treatment of raw juices at temperatures below 195°F. may provide some protection against clarification and spoilage during processing; however, for good stability in concentrates to be stored at temperatures from 32°-45°F., complete inactivation of pectinesterase is necessary.

SUMMARY

Heat treatment of Pineapple orange juice to 165°F. in three seconds prior to concentration to 42°Brix prevented clarification and gelation in the concentrate during storage at 80°F. for 24 hours; the microorganism count in the raw juice was also greatly reduced and approximately 75% reduction in pectinesterase activity was obtained.

To prevent clarification in hot-packed Pineapple orange concentrate during storage at 32°F., complete inactivation of pectinesterase by heat treatment is necessary.

LITERATURE CITED

1. Atkins, C. D., Wenzel, F. W., and Moore, E. L. An evaporator of improved design for the concentration of citrus juices. **Proc. Fla. State Hort. Soc., 64:** 188-191. 1951.

2. Atkins, C. D., Rouse, A. H., Huggart, R. L., Moore, E. L., and Wenzel, F. W. Gelation and clarification in concentrated citrus juices. III. Effect of heat treatment of Valencia orange and Duncan grapefruit juices prior to concentration. Presented at the Twelfth Annual Meeting of the Institute of Food Technologists, Grand Rapids, Michigan. June, 1952.

3. Huggart, R. L., Moore, E. L., and Wenzel, F. W. The measurement of clarification in concentrated citrus juices. **Proc. Fla. State Hort. Soc., 64:** 185-188. 1951.

4. Olsen, R. W., Huggart, R. L., and Asbell, Dorothy M. Gelation and clarification in concentrated citrus juices. II. Effect of quantity of pulp in concentrate made from seedy varieties of fruit. **Food Technol. 5:** 530-533. 1951.

5. Rouse, A. H., and Atkins, C. D. Heat Inactivation of pectinesterase in citrus juices. **Food Technol. 6:** 291-294. 1952.

PECTINESTERASE RETENTION IN CITRUS JUICES STORED AT VARIOUS TEMPERATURES[1]

A. H. ROUSE

Florida Citrus Experiment Station

Lake Alfred

During the past several years, this laboratory has conducted various investigations concerning pectinesterase in citrus fruits and juices (3, 4, 5, 6, and 7). Very little information is available in the literature on the effect of storage temperatures upon the activity of this enzyme, which is found in most fresh fruits and their juices. Hills and Mottern (1) have shown that tomato pectinesterase stored for one month at 32°F. lost 1% of its activity at pH 4.0, and when stored at 73.4°F. it lost 0.42% of its activity per day. MacDonnell, Jansen, and Lineweaver (2) reported on the effect of pH on the stability of citrus pectinesterase in salt-free solution and in borate-acetate extraction medium at 41°F. for two weeks. In the absence of salt most of the activity loss occurred in the first ten hours with very little activity being lost thereafter; whereas in the borate-acetate solution most of the decrease in activity occurred in two hours or less. However, in the latter case there was a favorable effect on stability at pH 6.0 and a deleterious effect below this pH. As the temperature was raised to 86°F., pH 7.5, the activity decreased gradually and inactivation was complete in two weeks.

Preliminary observations indicated that loss of pectinesterase activity occurred in citrus juices at room temperature. Therefore, this investigation was undertaken to determine enzyme losses in orange and grapefruit juices at their natural pH after storage at 80°F., 40°F., and —8°F. The results obtained should be of importance to the analyst interested in assaying citrus juices for their pectinesterase

[1] Cooperative research by the Florida Citrus Experiment Station and Florida Citrus Commission, aided by a grant from the American Can Company. Florida Agricultural Experiment Station Journal Series No. 102.