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# 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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these tests was supplied by a commercial plant, and had been prepared by conventional methods of low pressure evaporation, described in the literature (8).

The grapefruit concentrate was prepared from Duncan variety fruit in May, 1947. The juice was extracted in Rotary juice presses, strained through a 00 screen, and held in storage without pasteurization. The unpasteurized juice was continuously fed to a circulating evaporator where it was heated to 105 degrees F and allowed to expand in a vacuum of approximately 29.6". The juice was recycled a sufficient number of times to raise the soluble solids content to 56 percent. The concentrate was then passed through a Mallory sterilizer, attaining a temperature of 240° F. for one second. The heated concentrate was transferred to a vacuum expansion chamber and flash cooled to 160° F. The cooled concentrate was then filled into 96 oz. (No. 10) containers, sealed under vacuum, cooled to room temperature in a conventional cooler, dried, and placed in cases. The cased product was then transferred to cold storage (450° F. or lower.) (9)

The lime concentrate was supplied in unpasteurized, frozen form. This product was prepared by a commercial process described in the literature (10). One pound of sucrose was added per gallon of raw lime juice, prior to concentration.

The analysis of the concentrates on the date of receipt by the project

| | Orange | Grapefruit | Lime |
|---|---|---|---|
| Percent soluble solids | 64.23 | 61.42 | 51.52 |
| Percent titratable acid (as citric) | 4.50 | 4.69 | 24.30 |

Soluble solids were determined by the method of Stevens and Baier (11). Titratable acidity were determined by Official Methods of the Association of Official Agricultural Chemists (12).

Essential oils were furnished to the project by several processors. The oils utilized by the project:

a. Oil of orange, cold pressed, Floridian
2. Oil of tangerine, vacuum steam distilled, Floridian
c. Oil of lemon, cold pressed, Californian
d. Oil of grapefruit, vacuum steam distilled, Floridian
e. Oil of grapefruit, cold pressed, Floridian
f. Oil of lime, distilled and concentrated to five-fold, source unknown.

The above oils were submitted to J. W. Kesterson, Associate Chemist, University of Florida, Citrus Experiment Station, Lake Alfred, for analysis and concentrating. Representative samples of each oil (excepting lime were concentrated by Kesterson for the project.

## Experimental

A preliminary stock base was first developed, from which variations in essential oils, emulsifying agents, preservatives, etc., could be studied.

The amount of concentrate used in making the base was predicated on a final beverage containing 30 percent soluble juice solids, and employing approximately equal volumes of orange and grapefruit concentrates. Additional sucrose and citric acid were necessary to satisfy the total soluble solids and ratio requirements of the beverage.

The formula for one gallon of this beverage base without the addition of flavoring oils:

34 fl. oz. concentrated grapefruit juice.
34 fl. oz. concentrated orange juice.
1.73 lbs. sucrose.
0.31 lb. citric acid.
29 fl. oz. water.

This base, when diluted with an equal volume of 32° Beaume (59.1° Brix) sucrose syrup, and further diluted (or "thrown") with 5 volumes of water, yielded a beverage of the following calculated composition:

Degrees Brix.....11.9
Percent titratable acid (citric)....0.48
Percent citrus juice.....30.0

Initial studies with essential oils were based on a maximum oil content of 0.03 percent in the finished beverage. It was soon found on dilution that this amount was entirely too great, and it was reduced until a taste level satisfactory to the authors was attained. This level was finally found to be 0.0026 percent.

After establishing this taste level, attention was paid to the character of the flavor attained by varying the essential oils. It was soon noted that an orange-flavored drink could be made from the above stock base, by using only orange oil, but there was nothing distinctive to the product, since the objective of the work was to make a beverage with a *unique* flavor representative of Florida citrus. Combinations of oils were then tried. Combinations of orange oil with grapefruit oil did not prove satisfactory, grapefruit oil adding nothing to the flavor, and the oil mixture deteriorating rapidly.

The next oil combination tried was orange and tangerine. This mixture showed promise in lending an unusual flavor to the product. However, all tasters could detect the grapefruit concentrate character in the product, and did not care for that part of the flavor. Lemon oil was blended with the orange and tangerine oils, but the lemon oil masked the orange fraction of the flavor.

The final, successful, combination was a blend of orange, tangerine, and lime oils. After proper adjustment of the ratio between the three oils, a desirable flavor type was obtained.

The lime oil seemed to mask the grapefruit character of the drink, and in combination with the orange and tangerine oils, contributed a unique flavor. Project personnel and others commented that the flavor was distinctively citrus in character, but no one of the three flavor components could be selected as outstanding. This combination seemed to have the most promise in accomplishing the objective insofar as flavor was concerned.

After establishment of the ratio of orange, tangerine, and lime oils to produce the flavor type most acceptable to a taste panel, studies were undertaken to determine the advantages—if any—of employing concentrated oils. Three, five, and ten fold concentrations of the orange oil were prepared; three and five fold concentrations of the tangerine. The five fold oils were selected as superior to the others. Comparison by taste panel of beverages prepared from single strength and five fold oils indicated a preference for the concentrated oils. Insufficient data have been collected on the storage stability above 44° F. of bases prepared from single strength and concentrated oils to report at this time. It has been demonstrated, however, that minimum flavor change has occurred in storage at 40° F. or less, over the 4 month period the project has been underway.

The blend of concentrated oils finally selected as most desirable was composed of:

38 parts five-fold orange oil, Floridian.
19 parts five-fold tangerine oil, Floridian.
1 part five-fold lime oil.

1.2 ml. oils, blended as above were employed per gallon of beverage base.

In all of the experiments with oils the tasters commented that the drinks lacked "fruity" character. The type of flavor noted above seemed to be the most desirable, but it was not "rich" or "fruity" enough. Commercial beverage base often includes the use of salt to develop flavor, and this use has been noted by Jacobs (18). Salt has also been used in soft drink beverages to increase the sweetness of sugar solutions, as noted by Jacobs (14) and Dunn (15). Sodium chloride was therefore incorporated in the beverage base in an amount equivalent to 0.15 percent in the finished beverage. A decided improvement in flavor was noted. This was checked several times during the course of

the studies, by comparison of beverages. All tasters selected the beverage containing salt as superior in flavor to the one without.

Further improvement in the beverage was attained by the incorporation of frozen concentrated lime juice. An amount of lime concentrate sufficient to satisfy 5% percent of the added citric acid requirement was incorporated into the base. This amount of lime concentrate definitely improved the "fruity" character of the flavor of the beverage. Shelf-life studies over a two week period at room temperature (80-90° F.) indicated no flavor change or loss of "cloud." This beverage was given preference over all others by a taste panel.

Flavor enhancement was also noted in a series of studies made with surface active agents employed to disperse the oils. The orange-tangerine-lime mixture was dispersed with the aid of *lauryl pyridnium chloride*. This material was furnished to the project in the form of a solution containing 30 percent active L.P.C. thirty-six ml of the oil mixture were blended with 10ml of lauryl pyridinium chloride and 250 g dextrose. The blend of oils, dispersing agent and sugar was placed in a Waring Blendor and the mixer turned on. Sufficient distilled water was rapidly added to effect incorporation of the ingredients. Distilled water was further added to the suspension to give a final volume of 800 ml. This suspension was used to dose a quantity of beverage base in an amount sufficient to furnish the required concentration of oils. The amount of surface active agent (on an active ingredient basis) in the ultimate finished beverage was 0.00019 percent.

Beverages were submitted to a taste panel, representing (1) the basic oil mixture emulsified with gum acacia and (2) dispersed with lauryl pyridinium chloride. Two beverages were prepared for each agent; one containing salt, and one with salt withheld. The tasters selected the beverage containing lauryl pyridinium chloride and salt as the one with the most desirable flavor, commenting that the flavor was more "fruity." However, all tasters noted an "astringent" after taste.

Similar studies were performed with another surface active agent, alkyl dimenthylbenzylammonium chloride. This product had a flavor of its own which could be detected in all dilutions employed in the experiment. Further studies with surface active agents as dispersing agents for citrus oils have been deferred. These products have not been approved for use in foods by the Food and Drug Administration.

During the period of examining essential oil blends for suitability, many studies were made with regard to the efficacy of various emulsifying agents. Pectin was originally employed, in the forms of dry powder and a stock solution. An aqueous mixture containing 3 percent 100 grade citrus pectin proved to be satisfactory in producing an emulsion containing 15 percent of essential oils. The resultant emulsion—and all others containing pectin or gums—was homogenized by passage through an Eppenbach QV 6 colloid mill, utilizing minimum operating clearance between rotor and stator. A less satisfactory emulsion was prepared by dispersing dry pectin in the oils, before adding the aqueous phase. The stability of such emulsions was not uniform, and the thorough emulsification of the oils was dependent on the operator's ability to add the aqueous phase at a correct rate.

Gum acacia proved to be an efficient emulsifier. Here again, stable emulsions were consistently prepared when a stock aqueous solution of the gum was employed. Following recommended procedure for a "cloudy" type orange emulsion (16), 16 ounces of gum acacia were dissolved in 16 ounces of water, the solution filtered, and used to emulsify 0.75 ounces of blended oils. An emulsion volume sufficient to satisfy the oil requirements of the beverage was added to the base, substituting this amount for part of the water component of the formula. No significant increase in the "cloud" of the finished beverage was noted by the authors.

As a result of techniques employed in the handling of surface agents as dispersants for oils, a method of utilizing gums in the dry state was developed which resulted in consistently stable emulsions. An amount of dextrose sufficient to absorb the essential oils and present a "dry" mix was employed. After determining the amount of gum required, the gum and dextrose were "folded" together, and the essential oils blended in. This "dry" mix is then transferred to a high speed mixer (such as a Waring Blender), and while mixing, the aqueous phase is added in an amount sufficient to produce an emulsion of the consistency of milk. The resultant emulsion was then diluted to any required volume and homogenized. This technique has proved very successful in insuring complete emulsification of the oils.

Gum tragacanth proved to be superior to gum acacia as an emulsifier. Less tragacanth need be employed to obtain a stable emulsion. The usual amount employed in these studies was 5g, of tragacanth, 20ml oils, 50g dextrose, and water to make an emulsion volume of approximately 500 ml. When employing gum acacia, the amount was increased to 8-10 g.

Studies were undertaken to determine the effect of preservatives in varying concentrations. Sodium benzoate was incorporated into the base in an amount sufficient to give a concentration of 0.05 per cent in finished beverage. This concentration of preservative was detected by a majority of tasters. Lesser concentrations of preservatives were tried, but spotty spoilage developed in these concentrations. Therefore we do not recommend a concentration of preservative in the finished beverage of less than .05 per cent.

It is possible to market this product as a pasteurized beverage. Quite a number of bottling plants are now equipped to handle beverages in this form. If the product is intended for this particular purpose, no preservative need be included if storage of the beverage base at 40°F. or lower is strictly adhered to.

## Conclusion

Summarizing the work reported above, the following formula for one gallon beverage base incorporates the results of all the studies made thus far:

34 oz. orange concentrate
34 oz. grapefruit concentrate
11.0 oz. lime concentrate
784 grams sucrose
68 grams citric acid
32 grams sodium chloride
1.2 ml oil (in emulsion)
29 oz. water

Use: 1 gallon beverage base
1 gallon 32° Beaume sucrose syrup

Throw: 1 oz. to 6 oz. bottle

*Note*: The oils are blended in the ratio noted on page 6. Fifty ml of blended oils are mixed with 150g dextrose and 5g gum tragacanth and emulsified by the technique noted above. After the emulsion volume has been established and the emulsion homogenized, the amount of emulsion required to furnish 1.2 ml. blended oils per gallon of base can be determined.

The beverage has the unique flavor characteristic originally set as one of the goals for this project. It also uses a high proportion of grapefruit, another objective of the project. Finally, it opens for Florida citrus an outlet which should have worthwhile possibilities from an economic standpoint.

The authors wish to acknowledge the invaluable work of J. W. Kesterson, Associate Chemist with the University of Florida Citrus Experiment Station, Lake Alfred. The proper concentration and blending of the oils used would have been impossible without his full cooperation. The authors are also indebted to Citrus Concentrates, Inc., the Florida Citrus Canners Cooperative, the Vacuum Foods Corporation, and the Florida Citrus Oil Corporation for furnishing materials necessary to the project.

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# INTERNAL FRUIT QUALITY AS RELATED TO PRODUCTION PRACTICES

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It has been suggested that a practical paper covering the relationship between internal fruit quality and production practices would be appropriate at this time. An attempt has been made to keep the discussion simple and at the same time present in a general way what are considered good technical contributions by various research workers in the field. To try to discuss all of the factors which are reported by fact and fiction to influence citrus 'maturity" and fruit quality would be a hopeless task in the time alloted. This represents only a humble attempt to try and correlate some of what we know, plus a few logical assumptions, with some of the practical concepts about citrus "maturity" and fruit quality.

It is to be regretted that our present maturity standards do not serve as a better criterion of taste and flavor of citrus juice. It has long been recognized that the ratio serves little more than a satisfactory index of sweetness and sourness. Work by Cowart (2) of the Citrus Experiment Station, shows that at any given degree or sweetness, total solids is the best criterion of flavor. A juice with low solids is weak and flat and lacking in character while high solids gives juice character and richness or "body." The results of this work indicate that both artificially adjusted juice and natural orange juice having less than 8.8 percent solids is not acceptable to taste and has very little flavor regardless of the variety or acid content. Oranges with sol-