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The utilization and preservation of limes.

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THE UTILIZATION AND PRESERVATION OF LIMES

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THE UTILIZATION AND PRESERVATION OF LIMES
(Citrus Aurantifolia)

INTRODUCTION

In the United States limes are grown in conjunction with oranges, lemons, and grapefruit in Florida and California. They are also imported from Cuba, Mexico, and the West Indies where they grow wild on marginal or submarginal land.

Although the oil of imported limes is said to be of superior quality, the California and Florida fruits are leaders in flavor, keeping quality, and seedlessness. The harvesting of limes is carried on throughout the year but most pickings are made in the spring and summer. In the fall and the winter the few who want limes are willing to pay a premium for the California crop, which is then being harvested.

In North America, limes are chiefly consumed in the form of beverages. In Egypt, India and other foreign countries where limes are grown in abundance, they are employed in flavoring jellies, jams, marmalades, and as a garnish for fish and some meats.

Object of Study

Few reports have appeared in the literature pertaining to the composition and utilization of limes and

lime products. So far, this citrus fruit has been used largely for juice in the home. In this investigation it is proposed to study such lime products as jelly, marmalade, juice and lime peel oil. The vitamin C content of the lime and its juice will also be determined. This information may be useful to the lime grower, to the citrus by-product manufacturer, and to the consumer. With expanded production of limes in America, Asia, and Egypt there is need for studying a method of processing this fruit.

A REVIEW OF THE LITERATURE

The literature dealing with the manufacture of lime by-products is limited, as but little work has been done in this field. A summary of most of the pertinent information on this subject has been compiled.

Joslyn and Marsh (1939) reported that the lime resembles the lemon in structure and composition. It is higher in acidity than is the lemon and the lime-oil is higher in citral content than lemon oil.

McNair (1928) stated that lemon, grapefruit and lime juices tend to become bitter rapidly. None of these juices will ordinarily retain their natural flavors longer than twelve hours. Many methods have been used for the preservation of citrus juices such as pasteurization, sterilization by heat, ozone, sulphurous acid, carbon dioxide, benzoates or ultra-violet rays, together with the addition of sugar or by partial fermentation. The products, after being preserved by these different methods did not have the color, flavor or aroma of the fresh juice.

Cruess (1938) observed that lime juice rapidly developed a "terpeny" taste after bottling or canning. The juice was prepared by reaming the fruit, screening out seeds and coarse pulp, vacuumizing to remove oxygen, flash pasteurization, bottling at 175°F., sealing hot, and cooling. It was also prepared by flash pasteurizing, bottling warm,

sealing and pasteurizing in the bottle at 165°F.

Higby (1938) has found that the bitter taste and astringency, which develop in juices extracted from the immature form of both Valencia and Navel oranges after bottling or canning, are due to a substance called limonin which occurs in a non-bitter and water-soluble form in the albedo, the central fibrovascular bundles, and in the section covering of the fruit. When these tissues are ruptured the limonin is extracted into the juice where it is slowly converted to the intensely bitter lactone. The change from limonin to the bitter lactone form takes place slowly in the acid juice or in water with heating. Limonin has a molecular weight of 470 which corresponds with the formula $C_{26}H_{30}O_8$.

Higby (1941) further reported that in order to prevent the hydrolysis of limonin to the bitter complex the pH value of the juice must be increased. This was done by adding sodium bicarbonate, sodium carbonate, sodium citrate, or other buffers. The best results were obtained by the addition of enough lithium or sodium carbonate to the juice to neutralize from 15 percent to 20 percent of the total acidity, thus increasing the pH value to approximately 3.8 to 4.0. Carbonates helped, not only in preventing the hydrolysis of the limonin and iso-limonin to the bitter forming

complex, but also in dearating the juice by means of the carbon dioxide which was generated.

EXPERIMENTAL WORK

1. Composition of Limes and Lime Juice:

The chemical composition of whole limes and lime juice was determined* by methods of the Association of Official Agricultural Chemists (1940). The results are presented below in Table 1.

Table 1. Chemical composition of whole limes and lime juice

Component	Percent	
	Whole fruit	Juice
Moisture	77.49	90.23
Total carbohydrate	4.75	8.16
Protein	6.22	0.43
Fat	1.13	0.11
Fiber	11.20	--
Pectin	0.595	0.025
Ash	3.86	0.25
Total acidity (calculated as citric acid)	7.66	--
Calcium (content of ash)	0.58	0.02
Potassium (content of ash)	0.18	0.17
Calories per 100 grams	54.05	35.35

* The chemical analyses were made through the courtesy of Mr. P. H. Smith of the Feed Control Laboratory, Mass. Agric. Experiment Station.

2. Vitamin C (Ascorbic Acid) Content of Lime Juice

Studies were made to determine the vitamin C (ascorbic acid) content of lime juice. In this work determinations were made of the juice of limes grown in California, Florida, and Porto Rico. The ascorbic acid was determined by the dye titration method of Tillmans, Hirsch, and Hirsch (1932) as described by Maclinn and Fellers (1938). The 2,6-dichlorophenolindophenol dye solution was standardized according to the method of Buck and Ritchie (1938). A detailed procedure of these methods, as used, is presented below.

Preparation of indicator solution (0.05 percent):

1. Dissolve 0.2 gram of dry sodium 2,6-dichlorophenolindophenol in hot water, filter, and add water to make up to a volume of 400 ml.

2. Store in brown glass bottle - preferably in cold room - for not more than five days.

3. Standardize solution daily.

Preparation of potassium iodate solution (0.01 N.):

1. Dissolve 0.3567 gram of potassium iodate in warm water. Make up to one liter.
   Store in glass bottle. Keep covered to prevent evaporation as this is the standard solution.

Preparation of sodium thiosulphate solution (0.01 N.):

1. Dissolve 24.8 grams of sodium thiosulphate in distilled water and make up to one liter. This makes a 0.1 N. stock solution.

2. Dilute ten times to prepare a 0.01 N. solution as needed.

Preparation of starch solution (0.5 percent):

1. Dissolve 0.5 gram soluble starch in 100 ml. of cold water and store at a cold temperature.

Preparation of acid mixture (5 percent sulphuric acid and 2 percent metaphosphoric acid):

1. Dissolve 40 grams metaphosphoric acid in water and add 100 ml. concentrated sulphuric acid.

2. Make up to two liters.

Standardization of sodium thiosulphate against potassium iodate:

1. Place 2 grams potassium iodide in a flask and add about 50 ml. of water. Add 10 ml. of 0.1 N. hydrochloric acid and 10 ml. of potassium iodate.

2. Titrate against 0.01 N. sodium thiosulphate until a light straw color appears. Add starch indicator, five to ten drops.

3. Titrate to the disappearance of the starch color.

4. Repeat titrations in duplicate.

Standardization of dye solution against sodium thiosulphate:

1. Place 2 grams potassium iodide in a flask and add 50 ml. of distilled water.

2. Acidify with 10 ml. of 0.1 N. hydrochloric acid.

3. Add 10 ml. of dye solution to the flask.

4. Titrate against standard sodium thiosulphate solution to a straw color. Add starch indicator.

5. Titrate to disappearance of starch color.

Preparation and titration of sample of fruit juice:

1. Place 5 ml. of the fruit juice in a flask.
2. Add 25 ml. distilled water and acidify with 5 ml. acid solution.
3. Titrate against the dye solution, using the first pink that fades in just 40 seconds as the end point.
4. Make duplicate titrations.

The results of these determinations are summarized in Table 2.

Table 2. Ascorbic acid (vitamin C) content of fresh lime juice

Sample	Ascorbic acid per ml.
	mg
Juice from California limes	0.41
Juice from Florida limes	0.40
Juice from Porto Rico limes	0.34

From Table 2 it may be seen that fresh lime juice is a rich source of vitamin C. It compares very favorably in vitamin C content with the juice of other citrus fruits such as grapefruit, lemons, and oranges.

3. The Carotene (Vitamin A) and Riboflavin (Vitamin B_2) Content of Whole Limes and Lime Juice

The samples of whole limes for carotene and riboflavin analysis were prepared by cutting the fruit into small pieces and drying to constant weight at 140°F. The final results are calculated on a dry basis. The samples of juice were prepared by reaming the fruit, screening the extracted juice and holding it in brown bottles in a refrigerator until analyzed.

The carotene was determined by the method described by the Association of Official Agricultural Chemists (1940), using a Universal Spectrophotometer. Riboflavin was determined by the method of Hodson and Norris (1939). These results are shown in Table 3.

Table 3. Carotene and riboflavin content of whole limes and fresh lime juice

Sample	Carotene per gram	Riboflavin per gram
	micrograms	micrograms
1. Unscreened juice	0.30	0.27
2. Screened juice	0.30	0.19
3. Whole limes (dried basis)	6.00	4.00

From this data it may be seen that the carotene in fresh lime juice is not destroyed during the screening process. There was some loss of riboflavin at this stage of the juice preparation. There is also an indication that the riboflavin in lime juice is less stable during drying than is carotene. In general it may be considered that limes and lime juice are not good sources of carotene and riboflavin.

4. Lime Marmalade, Jelly, and Juice.

For many years citrus growers and some agricultural industries have been confronted with the problem of a satisfactory disposal of their surplus and cull material. During the last several years a large amount of money has been spent in experimental work on the utilization of by-products of the citrus fruits. In general, the results of this work have not been successful. A displeasing bitterness which develops during and after subjecting the juice to heat is one of the principal juice defects.

The present investigation of lime by-products was begun in July, 1941. It was found that as soon as the lime slices or juice were subjected to heat they lost their color instantly and developed a bitter flavor. Considerable time was spent in studying methods of preventing the hydrolysis of limonin to its bitter lactone, and the subsequent development of a bitter flavor. Many chemicals were tried in

different proportions. It was found that 0.15 percent calcium carbonate and 0.2 percent sodium carbonate by weight were sufficient to prevent the bitterness and retain the color. In the following pages a full description is given for using this method in making lime marmalades, jellies and preserved juices.

a. Lime Marmalade

Lime marmalade was prepared in a number of different ways in order to observe the effect of treatment with sodium carbonate, potassium carbonate, and ratio of sugar to juice. This experimental work may be outlined as follows:

A. The whole fruit was cut into thin slices and treated by the following methods.

   1. Slices were cooked in water until tender.
   2. 0.2 percent sodium carbonate was added to the lime slices and they were cooked in water until tender.
   3. 0.15 percent calcium carbonate was added to the lime slices and they were cooked in water until tender.
   4. 0.2 percent sodium carbonate plus 0.15 percent calcium carbonate were added to the lime slices and they were cooked in water until tender.

B. Lime juice was extracted from limes by reaming, screened through cheese cloth, and diluted with

varying amounts of water. This diluted juice was added to the washed lime slices.

C. Ratios of sugar to diluted juice of 1:2, 1:2.5 and 1:3 were used. The amount of sliced fruit was constant in all cases.

D. All of the ingredients - cooked sliced limes, diluted juice, and sugar - were mixed together and cooked to a jelly point (219.5°F.). The product was packed hot into 6 oz. glass jars, sealed and allowed to cool. The products were examined after five weeks' storage at room temperature.

All of the pertinent information in regard to the preparation and quality of the marmalade samples as described above is summarized in Table 4. From this data it is evident that the addition of sodium carbonate was valuable in retaining a satisfactory color in the marmalade. The addition of calcium carbonate was effective in preventing the development of a bitter flavor. A combination of sodium and calcium carbonates was necessary to maintain a good color and flavor in the finished product.

In Table 4 it may be seen that sample III-C4 was definitely superior to the other samples as regards color and flavor. A practical method of preparing such a lime marmalade as this is described as follows:

Table 4. Description of Marmalade Sample

Sample	Treatment	Wt.of lime peel	Wt.of lime juice	Wt.of water added	Amt.of sugar added	Ratio of sugar to diluted juice	Quality of finished product after five weeks' storage	
							Flavor	Color
		gms	gms	gms	gms			
I A1	none	280	450	900	600	1:2	Poor	Poor
I A2	0.2% Na_2CO_3 added	280	450	900	600	1:2	Poor	Good
I A3	0.15% $CaCO_3$ "	280	450	900	600	1:2	Good	Poor
I A4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	900	600	1:2	Fair	Good
I B1	none	280	450	900	530	1:2.5	Poor	Poor
I B2	0.2% Na_2CO_3 "	280	450	900	530	1:2.5	Poor	Good
I B3	0.15% $CaCO_3$ "	280	450	900	530	1:2.5	Good	Poor
I B4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	900	530	1:2.5	Fair	Fair
I C1	none	280	450	900	430	1:3	Poor	Poor
I C2	0.2% Na_2CO_3 "	280	450	900	450	1:3	Poor	Good
I C3	0.15% $CaCO_3$ "	280	450	900	450	1:3	Good	Poor
I C4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	900	450	1:3	Fair	Fair
II A1	none	280	450	1350	900	1:2	Poor	Poor
II A2	0.2% Na_2CO_3 "	280	450	1350	900	1:2	Poor	Fair
II A3	0.15% $CaCO_3$ "	280	450	1350	900	1:2	Good	Poor
II A4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	1350	900	1:2	Fair	Fair
II B1	none	280	450	1350	720	1:2.5	Poor	Poor
II B2	0.2% Na_2CO_3 "	280	450	1350	720	1:2.5	Poor	Good
II B3	0.15% $CaCO_3$ "	280	450	1350	720	1:2.5	Fair	Poor
II B4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	1350	720	1:2.5	Fair	Fair
II C1	none	280	450	1350	600	1:3	Poor	Poor
II C2	0.2% Na_2CO_3 "	280	450	1350	600	1:3	Poor	Good
II C3	0.15% $CaCO_3$ "	280	450	1350	600	1:3	Fair	Poor
II C4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	1350	600	1:3	Fair	Fair

Table 4 (continued)

Sample	Treatment	Wt.of lime peel	Wt.of lime juice	Wt.of water added	Amt.of sugar added	Ratio of sugar to diluted juice	Quality of finished product after five weeks' storage	
							Flavor	Color
		gms	gms	gms	gms			
III A1	none	280	450	1800	1125	1:2	Poor	Poor
III A2	0.2% Na_2CO_3 added	280	450	1800	1125	1:2	Poor	Fair
III A3	0.15% $CaCO_3$ "	280	450	1800	1125	1:2	Fair	Poor
III A4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	1800	1125	1:2	Good	Good
III B1	none	280	450	1800	900	1:2.5	Poor	Poor
III B2	0.2% Na_2CO_3 "	280	450	1800	900	1:2.5	Poor	Good
III B3	0.15% $CaCO_3$ "	280	450	1800	900	1:2.5	Good	Poor
III B4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$	280	450	1800	900	1:2.5	Good	Good
III C1	none	280	450	1800	750	1:3	Poor	Poor
III C2	0.2% Na_2CO_3 added	280	450	1800	750	1:3	Poor	Good
III C3	0.15% $CaCO_3$ "	280	450	1800	750	1:3	Good	Poor
III C4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	1800	750	1:3	Excellent	Excellent
IV A1	none	280	450	2025	1234	1:2	Poor	Poor
IV A2	0.2% Na_2CO_3 "	280	450	2025	1234	1:2	Poor	Good
IV A3	0.15% $CaCO_3$ "	280	450	2025	1234	1:2	Good	Poor
IV A4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	2025	1234	1:2	Good	Good
IV B1	none	280	450	2025	990	1:2.5	Poor	Poor
IV B2	0.2% Na_2CO_3 "	280	450	2025	990	1:2.5	Poor	Good
IV B3	0.15% $CaCO_3$ "	280	450	2025	990	1:2.5	Good	Poor
IV B4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	2025	990	1:2.5	Fair	Fair
IV C1	none	280	450	2025	825	1:3	Poor	Poor
IV C2	0.2% Na_2CO_3 "	280	450	2025	825	1:3	Poor	Fair
IV C3	0.15% $CaCO_3$ "	280	450	2025	825	1:3	Good	Good
IV C4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	2025	825	1:3	Good	Good

Table 4 (continued)

Sample	Treatment	Wt.of lime peel	Wt.of lime juice	Wt.of water added	Amt.of sugar added	Ratio of sugar to diluted juice	Quality of finished product after five weeks' storage	
							Flavor	Color
		gms	gms	gms	gms			
V A1	none	280	450	2250	1350	1:2	Poor	Poor
V A2	0.2% Na_2CO_3 added	280	450	2250	1350	1:2	Poor	Good
V A3	0.15% $CaCO_3$ "	280	450	2250	1350	1:2	Good	Poor
V A4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	2250	1350	1:2	Fair	Fair
V B1	none	280	450	2250	1080	1:2.5	Poor	Poor
V B2	0.2% Na_2CO_3 "	280	450	2250	1080	1:2.5	Poor	Good
V B3	0.15% $CaCO_3$ "	280	450	2250	1080	1:2.5	Good	Poor
V B4	0.2% Na_2CO_3 plus 0.15% $CaCO_3$ added	280	450	2250	1080	1:2.5	Fair	Fair
V C1	none	280	450	2250	900	1:3	Poor	Poor
V C2	0.2% Na_2CO_3 "	280	450	2250	900	1:3	Poor	Fair
V C3	0.15% $CaCO_3$ "	280	450	2250	900	1:3	Fair	Poor
V C4	0.2% Na_2CO_3 plus 0.15 $CaCO_3$ added	280	450	2250	900	1:3	Fair	Fair

1. Six limes are cut into thin slices and sufficient water to cover the sliced fruit during cooking is added. In addition 0.15 percent calcium carbonate and 0.2 percent sodium carbonate are added. The mixture is cooked for one and one-half hours, after which the slices should be washed thoroughly with cold water.
2. Juice from six limes is extracted by reaming, screened and then diluted with four volumes of water. The volume of juice before dilution should be 15 fluid ounces.
3. The diluted juice is added to the slices, then one pound and ten ounces of sugar are added and the mixture cooked to the jelly point.

b. Lime and Orange Marmalade

1. Four limes and two oranges were cut into thin slices, sufficient water, 0.15 percent calcium carbonate and 0.2 percent sodium carbonate were added. The slices were cooked for one and one-half hours after which they were washed thoroughly with cold water.
2. Juice from six limes was extracted by reaming, and screened. The amount of juice was 15 fluid ounces. It was diluted with four volumes of water.

3. The dilute juice was added to the cooked slices. One and three-eighths pounds of sugar were also added.
4. The combined fruit juice and sugar was cooked to the jelly point (219.5°F.), packed in jars, sealed, and stored for five weeks.

Observations were made at the end of the storage period. This product had a very good flavor and no objectionable bitterness.

c. Lime, Orange and Grapefruit Marmalade

1. Three limes, one-half a grapefruit, and one orange were cut into thin slices, sufficient water to cover the slices, 0.15 percent calcium carbonate and 0.2 percent sodium carbonate were added. The slices were cooked until tender. They were then washed thoroughly with cold water.
2. Juice from six limes (15 fluid ounces) was extracted by reaming, screened through cheese cloth and then diluted with four volumes of water.
3. The amount of sugar added to the slices and the diluted juice was one and three-eighths pounds.
4. The mixture of fruit, diluted juice, and sugar was cooked to the jelly point (219.5°F.), packed in six ounce jars, sealed, and stored for five weeks.

This product had a very good flavor and no objectionable bitterness.

5. Lime Jellies

a. Lime Jelly

A. The juice from 24 limes was extracted by reaming, diluted with four parts of water, and 0.2 percent by weight of calcium carbonate was added.

B. This juice was mixed with two and one-half pounds of sugar and then heated for ten minutes, during which time ten grams of 100 grade citrus pectin were added.

C. This jelly stock was then divided into three parts:

1. The first portion was filtered through four layers of cheese cloth.

2. The second portion was filtered with suction through "Filter-Cel" held in a Buchner funnel by circular piece of canton flannel covering the funnel bottom.

3. The third portion was treated with clarex (an enzyme preparation). The clarex acts as a clarifying agent and precipitates colloidal material in the juice. The clarex was prepared by mixing it with a small amount of the jelly stock and heating for 50 minutes at 100°F. The jelly stock to be treated was placed in a pan and its temperature raised to 76°F. One-tenth percent by weight of clarex (in the prepared form) was added and the jelly stock held at 76°F. for 24 hours. At the end of this time the mixture was heated to 212°F. to kill the enzymes. It was then filtered through Filter-Cel.

D. Each of the above three portions of jelly stock were cooked to the jelly point (219.5°F.), filled hot into glass jars, sealed, and stored for four weeks.

At the end of four weeks the samples were examined. Portion 1 as prepared above was very cloudy, while portions 2 and 3 were very clear. All of the samples had a good flavor and no objectionable bitterness. However, they did have a very strong acid taste.

b. Orange and Lime Jelly

In making this jelly the same procedure as in portion 1 (above) for lime jelly was followed except that proportions of lime juice to orange juice of 1:1 and 1½:1 and 2:1 were used. It was not necessary to use added pectin. These jellies had an excellent flavor.

c. Lime and Apple Jelly

The same procedure was followed in portion 1 (above) for lime jelly with the exception that proportions of lime juice to apple juice of 1:1, 1½:1, and 2:1 were used. The apples were peeled, sliced, cooked with small amount of water until tender, and the juice strained off through cheese cloth.

The lime-apple jellies had a very good flavor. No objectionable bitter flavor was present in them.

6. Vitamin C Content of Lime Marmalades and Jellies.

The different samples of lime marmalades and jellies were analyzed for vitamin C by the method previously described. In addition to vitamin C, their soluble solids

contents were determined with an Abbe refractometer, and total acidity (as citric acid) was determined. A Beckman pH Meter was used to measure their pH value. These analytical results are summarized in Table 5.

Table 5. Vitamin C (ascorbic acid) content of lime marmalades and jellies

Product	Ascorbic acid per gram	Total acids as citric	Soluble solids content	pH value
	mg	percent	percent	
Lime marmalade	.008	1.54	65	2.54
Lime and orange marmalade	.100	1.48	66	2.95
Lime, orange, and grapefruit marmalade	.020	1.52	65	2.60
Lime jelly	.023	1.90	62	--
Lime and orange jelly	.041	1.70	66	2.65
Lime and apple jelly	.029	0.91	--	--

From the above information it may be seen that the lime marmalades and jellies prepared in this investigation were relatively low in ascorbic acid content and cannot be considered as good sources of vitamin C. With the exception of the lime and apple jelly all of the products were characterized by a relatively high total acidity.

7. Preserved Lime Juice

The fresh flavor of lime juice is quite unstable, and it loses its natural flavor after twelve hours. It is recognized that two of the factors which affect the flavor and cause bitterness in the lime juice are:

1. The presence of air contaminated with organisms causing spoilage. (McNair, 1926)

2. The hydrolysis of the limonin and iso-limonin to the bitter lactone which takes place slowly in the acid juice or in water with heating. (Higby, 1938)

In this investigation several methods were tried in order to obtain a preserved lime juice which maintained its original fresh flavor and remained free from bitterness. One of the methods used consisted of packing the juice in bottles and using sodium benzoate as a preservative. This method was not satisfactory. It was also found that bottling the lime juice and sterilizing it by heat or flash pasteurizing it at 175°F. or 190°F. were not successful methods, from the standpoint of flavor retention. However, it was found that lime juice can be preserved for a long time at room temperature, without losing its flavor and without developing objectionable bitterness, by using the following method:

1. Green mature fruits were washed, cleaned, and cut in halves.

2. The juice was extracted by reaming and screened through a dry, clean cheese cloth in order to remove the pulp and seeds which, if mixed with the juice, would ruin its flavor.

3. Calcium carbonate in a concentration of 0.15 percent was added to the juice.

4. The juice was flash pasteurized immediately at 165°F. It was then filled hot into bottles, vacuum sealed, and cooled immediately.

Some of the sealed bottles were stored at room temperature (75°F.) while others were held in a refrigerator at 40°F. After four weeks' storage the samples had a good flavor, and were free from objectionable bitterness.

After four weeks' storage the lime juice samples stored in the refrigerator and at room temperature had a vitamin C (ascorbic acid) content of 0.39 and 0.36 mg. per gram, respectively.

8. Lime Juice Concentrate

The purpose of this study was to produce a concentrated lime juice having the characteristic color, flavor and food value of fresh juice.

Freshly screened juice was concentrated by using a constant temperature and a high vacuum by the method described by Sedky, Fellers and Esselen (1942). The apparatus used for this experiment is shown in Plate I.

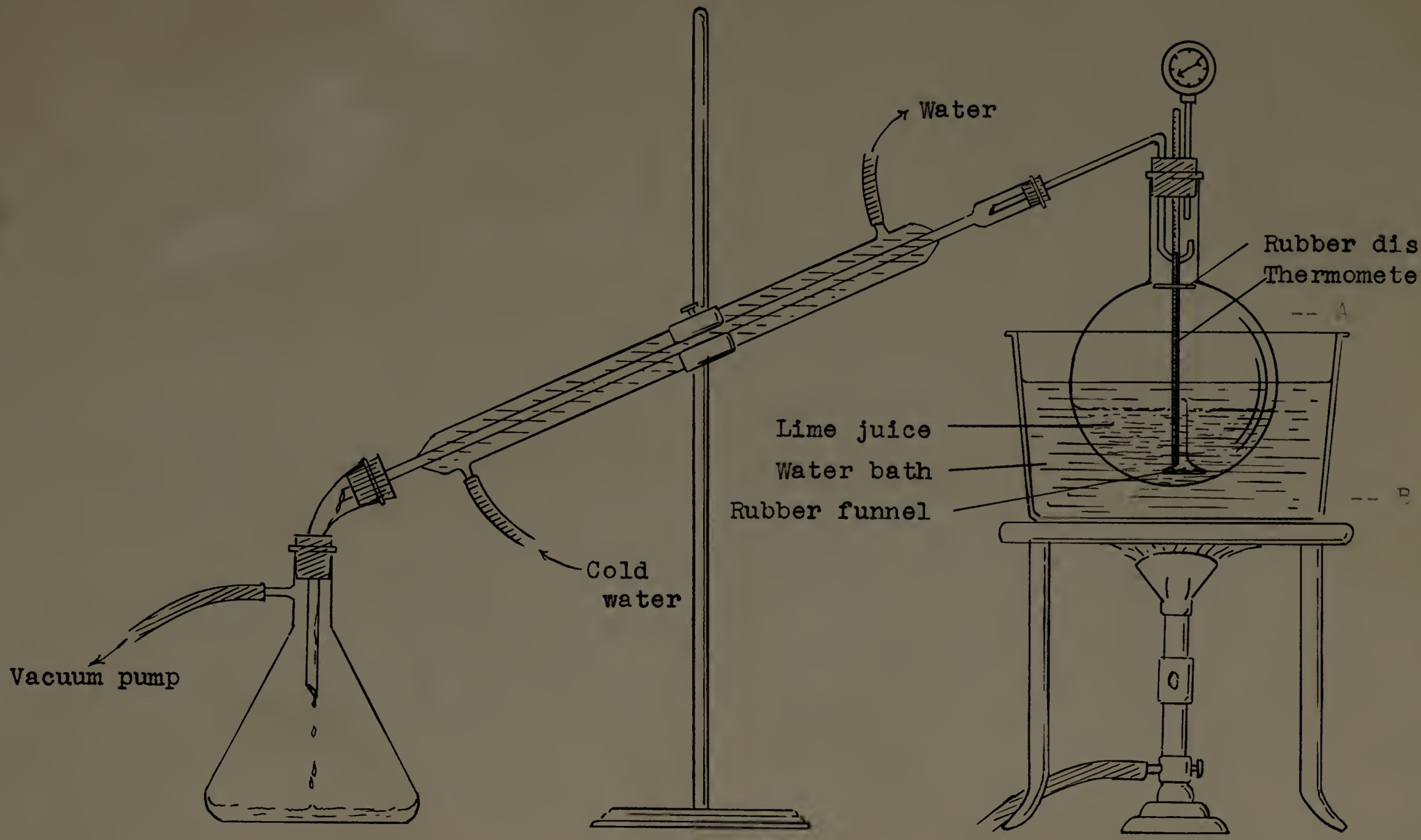

Water
Rubber disc
Thermometer
Lime juice
Water bath
Rubber funnel
Cold
water
Vacuum pump
A
B
C

Plate I

Procedure:

A. 1000 ml. of fresh screened lime juice was put in the flask (A) and 0.2 percent of calcium carbonate was added.

B. The concentration was carried on at constant temperature by adjusting the flame under water bath (B).

C. During concentration a vacuum of 25 inches of mercury was maintained in the apparatus, while the temperature was 100°F.

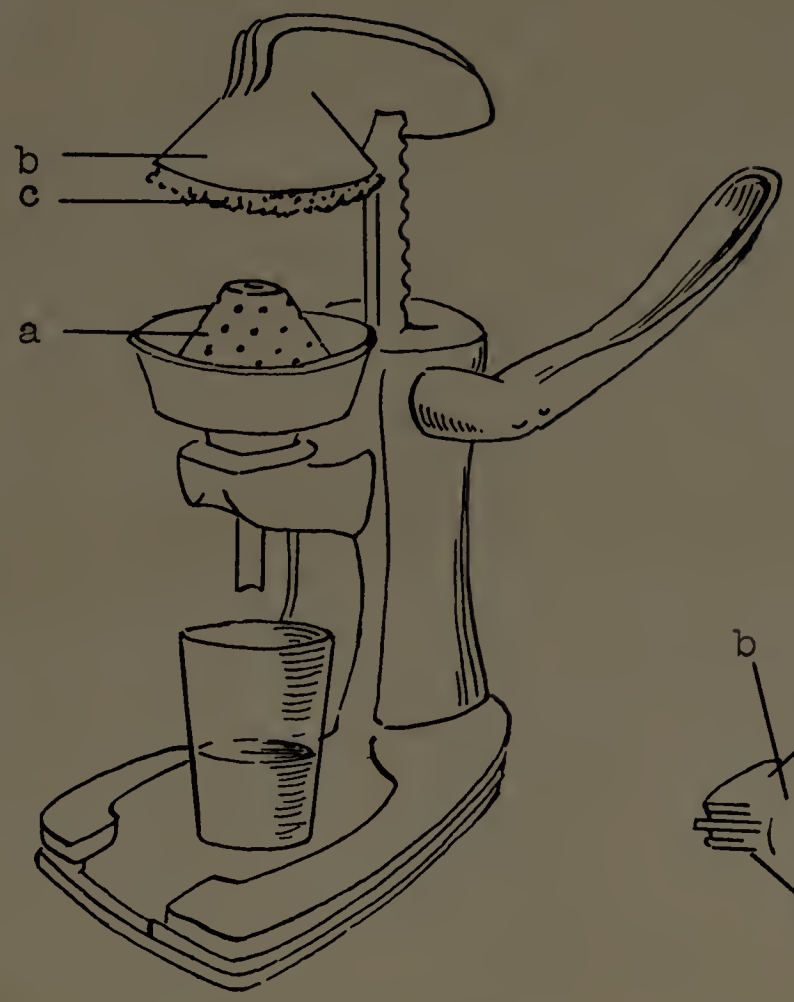
D. The concentrated juice was filled into bottles, capped, and pasteurized in a hot water bath until the temperature of the product reached 165°F.

After three weeks' storage at room temperature it was found that the samples had a good flavor and were free from objectionable bitterness.

The quantity of lime juice concentrate secured from one thousand milliliters of fresh juice, its vitamin C content and acidity are presented in Table 6.

Table 6. Yield and composition of lime juice concentrate

Sample	Original volume of juice	Amt. concentrated juice obtained	Vitamin C content per ml.	Total acidity (as citric acid)	pH Value
	ml	ml	mg	percent	
1	1000	500	0.77	11.52	2.5
2	1000	350	1.12	15.97	2.5



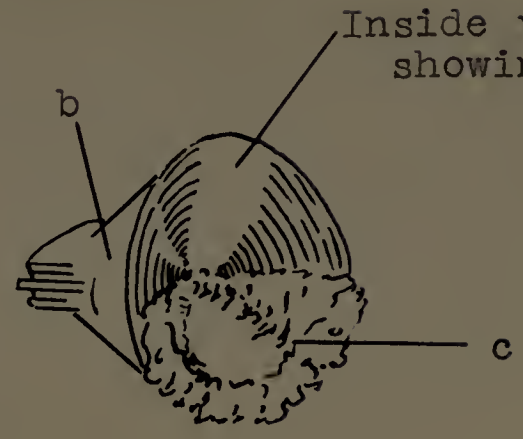


Plate II

9. Lime Oil

Methods of extraction

A. Extraction by expression

In limes as in other citrus fruits, the oils are found in the cells of the rind. Oil can be obtained by crushing these cells and absorbing it with small sponges.

It has been found that a fruit juice squeezer with a layer of sponges fixed in the upper part of it (Plate II) gives a good yield of oil. The whole fruit was cut in halves, which were held in the inverted cup (a) and pressed against the metal form which had the same shape as the cup (b), and was covered from inside with a layer of sponges (c). Upon pressing, the sponges absorbed the oils while the juice was extracted. The pressed halves were then pressed again in order to obtain most of the oil from the rind.

The chief advantages of this method are its simplicity and the fact that large quantities of material can be handled at small cost.

B. Extraction by steam distillation

The cleaned peels were cut by a revolving food chopper into pieces, transferred to a round bottomed flask (a), (see Plate III) and sufficient water was added to cover them. The flask was connected to the condenser and live steam was then drawn through the suspended peel by a glass

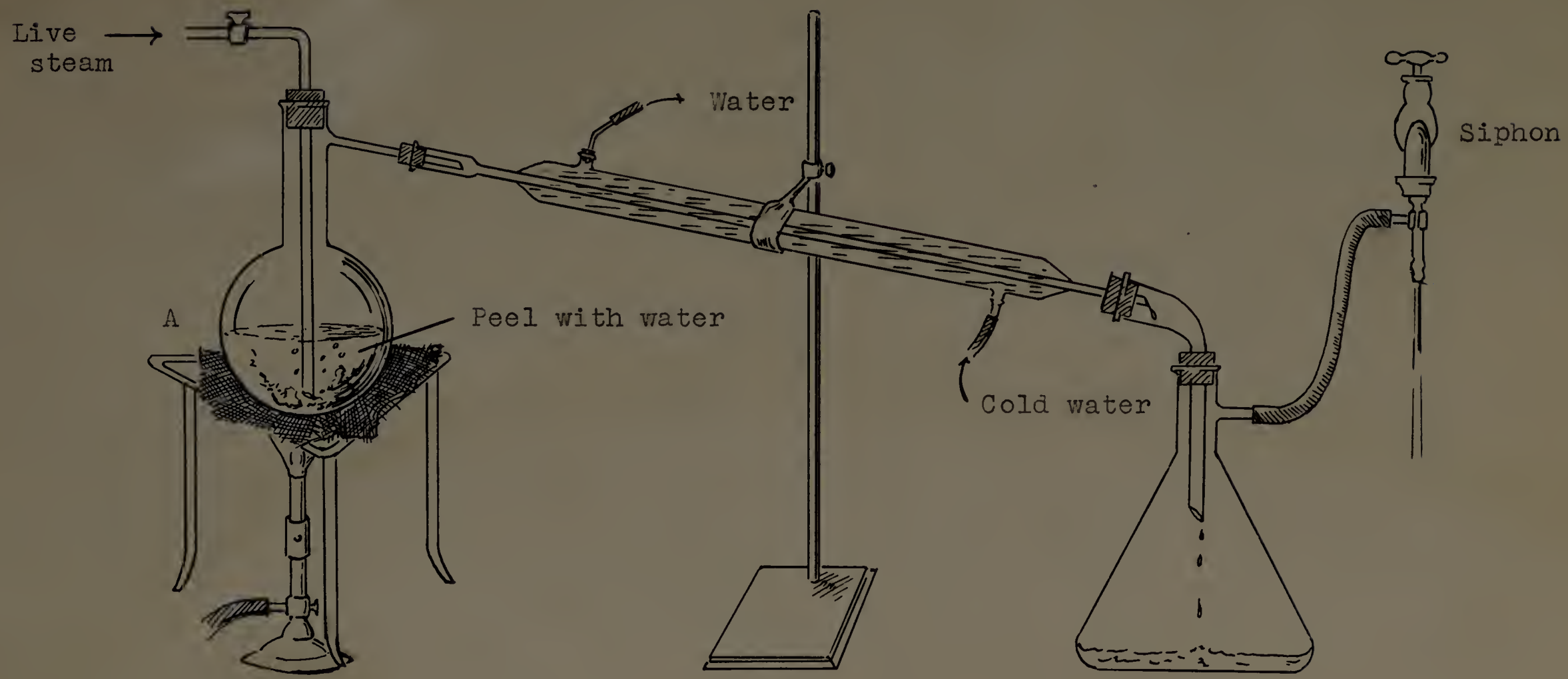


Plate III

tube (b). The distillate was collected in a flask connected with a water pump by which a slight suction was secured in order to draw the live steam and reduce the pressure. Steam distillation was continued until the condensate was free from turbity. This point indicated that the oil was entirely removed from the peels.

The distillate was then transferred to a separatory funnel and allowed to stand until the oil separated. The remaining traces of oil were removed from the distillate by means of a super-centrifuge.

The samples from both methods of extraction were stored in dark bottles and dried by adding a small quantity of anhydrous sodium sulphate. It was found that a ton of limes should yield about ten pounds of oil.

DISCUSSION AND RESULTS

The ascorbic acid content of limes and lime by-products from different locations are shown in Table 7, as averages of all determinations:

Table 7. Ascorbic acid contents of limes and lime by-products from different locations

Where grown	Ascorbic acid in lime juice per ml.	Ascorbic acid in lime marmalade per gram	Ascorbic acid in lime jellies per gram
	mg	mg	mg
California	0.410	0.008	0.023
Florida	0.403	0.008	0.022
Porto Rico	0.344	0.007	0.022

These experiments were carried from July 1941 to March 1942. Variations with locality where grown are presumably due primarily to differences in soil and climate. In Egypt, fresh lime juice is most frequently consumed in the form of beverages such as "Lime-Ade", in which water and sugar are added. Tests conducted on lime juice when made into beverages showed no loss in its vitamin C content. Therefore a glass (300 cc.) of "Lime-Ade" made from the juice of one lime (43 cc.) would furnish about 14 mg. of vitamin C.

In order to determine whether ascorbic acid content was affected by cold storage, some fruits were placed in an ordinary cold storage for forty days. It was found that they showed no decrease in ascorbic acid content after storage. The greatest losses of ascorbic acid in marmalades and jellies occurred in the cooking of the peel. Addition of calcium carbonate also caused a decrease of ascorbic acid compared with the original materials.

Small amounts of calcium carbonate (.15 percent by weight) helped in preventing the hydrolysis of the limonin and iso-limonin. The hydrolysis of these two compounds results in the splitting of the molecule with the liberation of bitter substances. Higby (1941) reported that addition of lithium or sodium carbonate to the navel orange juice prevented the formation of the bitter complex. Sodium carbonate developed a sourness and did not prevent bitterness in lime juices. It can only be used in making marmalade as a means of retaining the color of the peel. The peel should be washed several times with cold water in order to eliminate the sourness developed by this substance.

SUMMARY

Lime juice is a rich source of ascorbic acid. The California limes contain 0.41 mg. per ml., while Florida and Porto Rico limes contain 0.40 mg. and 0.34 mg. respectively. That is, the lime is as good a source of vitamin C as oranges and grapefruit.

The amount of carotene and riboflavin in limes and lime juice is negligible.

The ash of limes is alkaline in character, consisting largely of calcium and potassium.

The presence of 0.15 percent calcium carbonate by weight is sufficient to stop the hydrolysis of the limonin to the bitter lactone in lime juice, marmalade, and jelly.

The addition of 0.15 percent of calcium carbonate by weight to the screened juice and the use of flash pasteurization at 165°F. for a few seconds aided in preserving the natural aroma and taste of the juice and its vitamin C potency, during storage in glass containers.

Calcium carbonate raises the pH value and helps in preventing the hydrolysis of the limonin and iso-limonin to the bitter lactone complex. It also helps in sweeping the juice free of oxygen by the carbon dioxide generated.

Lime juice was concentrated by using a constant temperature and a high vacuum. The concentrated juice was pasteurized at 165°F. and cooled. This concentrated juice when diluted gave a pleasant refreshing flavored product

which could be used in the manufacture of marmalade, jellies, sherbets, ices, etc.

Lime oil can be obtained by crushing the cells of the rind. Two methods have been used.

1. Extraction by expression.
2. Extraction by steam distillation.

Both methods gave good yields, although the first one is easier and cheaper. One ton of fresh limes yields about 9.5 pounds of oil.

BIBLIOGRAPHY

Association of Official Agricultural Chemists, 1940. Official and tentative methods of analysis of the association of official agricultural chemists. P.O.Box 540. Benjamin Franklin Station, Washington, D. C. 757 pp.

Bennett, A. H., and Tarbert, D. J., 1933. Vitamin C in citrus juices. Biochem. J., 27, 1294-1301.

Bhat, S. S., 1939. Indian lime juice industry, its present and future. Bibliog. pl. map. Agr. and Livestock, India. 9, 418-31.

Buck, R. E., and Ritchie, W. S., 1938. A new method for the standardization of the dye used for the determination of vitamin C. Anal. Ed. Ind. and Eng. Chem. 10, 26.

Camp, A. F., 1932. Citrus fruit juices. Proc. Fla. State Hort. Soc., 1932. p.60-65.

_____, 1933-A. Production of limes in Florida. Fla. State Hort. Soc., 1933, p.115-20.

_____, 1933-B. Study on the preservation of citrus juices and pulps. Fla. Agr. Exp. Sta. Ann. Rept., 1933, p.102-104.

Chace, E. M., 1920. A method for the clarification and preservation of fruit juices. Calif. Citrograph, 5, 264.

_____, 1925. By-products from citrus fruits. U. S. Dept. Agr. Circ. 232, Rev. 1-14.

Davies, S. M., 1921. Loss of citric acid in limes and lime juice. Agr. News 20-25.

Floyd, W. W., 1939. Changes in vitamin C content during boiling of turnip greens in various water in covered and uncovered containers. Food Res. 1940, p.33.

Hardy, F., and Warneford, F. H. S., 1925. Coloring matter of lime juice. Ind. Eng. Chem., 17, 48.

Higby, R. H., 1938. The bitter constituents of navel and valencia oranges. J. Am. Chem. Soc., 60, 3013-18.

_____, 1941. Canning navel orange juice. Calif. Citrograph, October, 1941.

Hodgson, R. W., 1920. Possibilities for lime culture in California. Calif. Citrograph, 5, 303.

Hodson, A. Z., and Norris, L. C., 1939. A fluorometric method for determining the riboflavin content of foodstuffs. J. Biol. Chem., 131, 621-630.

Joachin, A. W. R., 1927. Experiments of the preservation of lime juice at Peradeniya. Biblio. Tropical Ag. 69, 155-8.

Joslyn, M. A., and Marsh, G. L., 1934. Some factors involved in the preservation of orange juice by canning. Fruit Prod. J., 14, 43-49.

_____, 1937 A. Utilization of fruit in commercial production of fruit juices. Calif. Agr. Exp. Sta. Circ. 344, 63pp.

_____, 1937 B. Fruit juice canning. Food Industries, 9, 580-583.

Loeseche, H. W., 1933. Some problems in citrus products. Res. Proc. Fla. State Hort. Soc., 1933. p.38-43.

Lund, H., Spur, B., and Fridericia, L. S., 1934. The biological and titrimetric determination of vitamin C. Biochem. J., 28, 1825.

Maclinn, W. A., and Fellers, C. R., 1938. Ascorbic acid (vitamin C) in tomatoes and tomato products. Mass. Agr. Exp. Sta. Bul. 354, 39 pp.

McNair, J. B., 1926. Citrus products, Part I. Field Museum of Natural History Pub. 238. Bot. Ser. VI No. I, 392 pp.

Pope, W. T., 1923. The acid lime fruit in Hawaii. Hawaii Agr. Exp. Sta. Bul. 49, p.1-20.

Poore, H. D., 1932. Analyses and composition of California lemon and orange oils. U. S. Dept. Agr. Tech. Bul. 241, p.1-30.

Richardson, J. E., Davis, R., and Sullivan, P., 1937. Some observations on vitamin C content of oranges and lemons. Food Res., 2, 81-83.

Roberts, J. A., and Gladdum, L. W., 1937. Composition of citrus fruit juices. Ind. Eng., 29, 574-575.

Sedky, A., Fellers, C. R., and Esselen, W. B., Jr., 1942. An improved orange juice concentrate. Fruit Prod. J., 21, No. 5, p.136-138.

Spooner, A., 1930. Limes. Jamaica Ag. Soc., p.518-20.

Tillmans, J., Hirsch, P., and Hirsch, W., 1932. The reduction capacity of plant foodstuffs and its relation to vitamin C. Z. Untersuch. Lebensm. 63, 10.

Tressler, D. K., Joslyn, M. A., and Marsh, G. L., 1939. Fruit and vegetable juices. Avi Pub. Co., New York, N. Y. 549 pp.

Webber, H. J., 1932. The lime in California. Calif. Citrograph, 17, 456-7, 478-9, 18, 19, 32.

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Graduate Committee.

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