# EFFECT OF DIFFERENT PREPARATION TREATMENTS ON THE FLAVOR AND ASCORBIC ACID CONTENT OF MEXICAN LIME JUICE AFTER FROZEN STORAGE

by

HUSSEIN OSMAN AHMED OSMAN

A THESIS

submitted to

OREGON STATE COLLEGE

in partial fulfillment of
the requirements for the
degree of

MASTER OF SCIENCE

June 1951

APPROVED:

Professor of Food Technology

In Charge of Major

Head of Food Technology

Chairman of School Graduate Committee

Dean of Graduate School

Date thesis is presented December 2, 1950

Typed by Regina Long

ACKNOWLEDGEMENT

The writer wishes to express his gratitude to Dr. E. R. Parker and Dr. W. P. Bitters of the Citrus Experiment Station, University of California at Riverside, for providing the limes; to Dr. Oliver J. Worthington for his valuable help throughout the course of work and in preparing this manuscript; to Professor E. H. Wiegand for his interest and to the members of the taste panels and the Food Technology Department for their time and effort. Appreciation is due Botross Bey Bassili, Dr. J. L. Heid, Mr. E. L. Moore, and Dr. A. A. Moursi for kindly providing information through personal communications.

TABLE OF CONTENTS

# LIST OF TABLES

# LIST OF FIGURES

# CHAPTER I

## INTRODUCTION

The weather and soil conditions of Egypt favor the production of citrus fruits, which was known to the Pharaohs of Ancient Egypt (16). In the year 1949 (4), Egypt had 3,857 acres of cultivated acid lime orchards which produced 1,278,729 thousand limes, or about 52,000 tons.

Limes can be divided into two groups (33):

1. Sweet limes
2. Acid or sour limes.

The acid limes are divided into:

1. The true limes, or the Mexican group. The fruits are acid with very thin rind. It is considered to be the type of the species (Citrus aurantifolia).

2. The large fruited limes, or the Tahiti group. The fruit is much larger and almost totally seedless; the odor, although resembling that of the Mexican, is less pronounced; and the flavor, although fully as acid, does not have the same pronounced lime bouquet.

Limes of the Mexican group were used in this experimental work.

Dr. Moursi (23) states that there are at least three different varieties of sour limes in Egypt:

1. The Egyptian lime "Rashidi, Benzahair or Baladi". This is by far the most dominant variety. Its harvesting reaches its peak during the months of July, August, September and October. The fruit weighs about 34 grams; its rind is thin and pale yellow when ripe; its pulp is pale, juicy and acidic. A full grown tree may produce up to 2,000-3,000 fruits.

2. West India limes; This variety is grown on a small scale and was introduced from the West Indies in 1911. The fruit is comparatively small; it weighs only about 25 grams; the yield per tree is much smaller than that of the Baladi. The crop season of this variety is during the month of October.

3. Seedless lime, "Sultan Hussein": This variety was introduced from one of the Greek islands. The fruit is oval in shape and is about 85 m.m. in length and 48 m.m. in diameter; it is white in color, juicy and acidic. This variety is not very common.

Mexico and the West Indies also produce large quantities of limes (33). In the United States (33), lime culture extended rapidly during the decade beginning in 1930. It is, mainly, grown in Florida, California, and Arizona. Besides, some quantities are imported from the West Indies and Mexico.

From the previous data concerning limes in

Egypt, one can see that limes there are available during a part of the year while they are less prevalent during other seasons. This problem was always recognized and attempts to solve it were undertaken. The first attempt to be mentioned is the regulation of irrigation of lime trees (called in Egypt fasting) so it would give its product during the time in which the fruit is scarce. This is done fairly successfully in the province of Fayum, some 100 miles to the wouthwest of Cairo.

Another approach would be the cold storage of the limes. In this attempt, several difficulties were encountered not only in Egypt but also in other places. Aref (2) of the Food Technology Department, Fouad 1st University, Cairo, Egypt, recommends storage at 45° - 48° F. with a relative humidity never below 85% and ranging between 85% and 95%. He gives an approximate figure of eight weeks storage. Miller (21), who carried an intensive study on cold storage of citrus fruits including limes in Florida, recommends the same temperatures and relative humidity range, giving a storage duration time of six to eight weeks.

Still another approach could be the preservation of lime juice by canning or bottling, but the flavor of citrus juices is very susceptible to heat, a factor which eliminates this method. Very recently, however,

canned limeade has appeared on the American market. Limeade is, of course, limited to beverage purpose only, water and sugar being added.

In Egypt, chemical preservatives are sometimes used in bottling lime juice. Usually, sodium benzoate is the preservative used. The product is unsatisfactory (12) and an undesirable taste develops after a short storage period (27). At least one British company puts a bottled lime juice product on the market preserved with sulfur dioxide. When it was submitted to a taste test panel here, it scored unacceptable, and one taster reported it tasted like medicine.

Table 1, which is provided by the Department of Economics, Egyptian Ministry of Agriculture (4), shows considerable higher prices for acid limes during the months March, April, and especially May, 1949, in comparison with those of August and September of the same year. No prices are given for June and July, probably because of the insignificant amount of acid limes available during these two months.

Since there are several uses for fresh flavored lime juice besides as a beverage, the freezing preservation of lime juice would seem quite logical in view of fresh quality retention of citrus juices by freezing. The results of freezing experiments are reported and discussed in the following pages.

Table 1

Prices of Sour Limes in Egypt during 1949

| Month | Minimum | Maximum | Month | Minimum | Maximum |
|---|---|---|---|---|---|
| | 100/P.T* | 100/P.T* | | 100/P.T* | 100/P.T* |
| January | 9 | 12 | July | -- | -- |
| February | 10 | 10 | August | 5 | 13 |
| March | 18 | 25 | September | 5 | 13 |
| April | 25 | 30 | October | 7 | 15 |
| May | 35 | 65 | November | 7 | 20 |
| June | -- | -- | December | 15 | 20 |

*Piastre: One piastre is equal to 1/100 of an Egyptian pound. One Egyptian pound is equal to 2.87 dollars (October, 1950).

# CHAPTER II

## REVIEW OF THE LITERATURE

In 1938, M. A. Tempany (29) wrote "The preservation of citrus fruit juices in a natural condition is by no means a simple problem. Such juices, and particularly orange juices, are liable to undergo changes on storage which include not only ordinary fermentation, but also oxidation processes which result in loss of flavour and of colour. Moreover, it is not possible to treat them by sterilization or even normal pasteurization as this results in imparting to them a cooked flavour which is unpleasant and also destroys the vitamins."

This was written in a time when the industry of freezing orange juice was something new, but still it holds true.

Ordinary low-temperature pasteurization was the common process in preserving citrus juices. But since pasteurization (30) must be carried out under conditions which will not impair the flavor of the juice or impart a cooked taste to it, flash-heating was suggested (8) to overcome this difficulty. This process consists of heating the juice rapidly to a temperature of 190° - 205° F., holding at this temperature for 10 - 60 seconds, cooling to a suitable filling temperature, then filling

into suitable containers.

Although better results were obtained with this new method, yet the product was not satisfactory (15) and there are certainly many of us who still remember the cooked taste of orange juice preserved by this method. Experiments at this time were taking place on preserving citrus juice by freezing. Beginning from 1931, several papers (3, 6, 9, 13, 17, 19, 24, 34) appeared, introducing and dealing with freezing citrus juices. They were first frozen single strength but several factors (31) like difficulties in handling, high storage and shipment costs, and the long time required for thawing, besides spoilage difficulties encountered during thawing, necessitated concentrating the juice before freezing.

There are many different methods for concentrating juices (10), but the two which were known-- previous to 1948-- to employ lower temperatures were:

1. Concentration in vacuum (11, 25, 29)
2. Concentration by freezing (28, 29).

While the second method is not known to be used or to have been used on a commercial scale as a step in the production of concentrated frozen citrus juices, probably because of high costs and technical difficulties, the first method was and still is used to a

certain extent in this industry. However, this method encounters the use of heat at about 110° F. in concentrating the juice under high vacuum. Although the product was superior to any previous one, yet the investigators kept trying to devise a method for concentrating the juice at even lower temperatures. Heid and Beisel (15) introduced, in 1948, a new method for concentrating citrus juices (1). In this method, both sides of the refrigerant compressor system are used. Water is evaporated from the juice in one tubular heat exchanger with heat given up by condensing compressed ammonia. Vapors would be condensed in a second tubular heat exchanger by evaporating the liquid ammonia through an expansion valve. This method concentrates the juice at a temperature between 50° and 70° F. thus avoiding undesirable high temperatures. This refrigeration system method has gained great appreciation and many plants switched to its use. The product is very desirable and gained much favor with the consumer.

Oxygen is well known to be a source for vitamin and flavor destruction (9). Therefore, packing in absence of air, either by using vacuum or an atmosphere of inert gas, has been recommended (7, 29). $CO_2$ was reported to produce a carbonated taste at ordinary room temperature while nitrogen had a beneficial effect (7).

Chace and Poore (9), in introducing frozen citrus juices in 1931, reported, "So far as could be determined organoleptically, there was little difference in flavor between the juice frozen without preliminary treatment and that from which the air had been removed by $CO_2$. Excessive use of $CO_2$ had to be avoided, however, owing to the off-flavor developed."

It is well known that flavor, odor, color, texture and nutritive values of foods often diminish in the presence of oxygen. Walker of the Linde Air Products Co. (32) reports that, "Nitrogen prevents oxidation in foods by removing and excluding air from foods by displacement and/or protecting foods by blanketing them during processing and packaging." He states that many foods (especially liquids) are in equilibrium with air early in their preparation. Then he adds that nitrogen, carbon dioxide, or steam may be used as a purging gas under certain circumstances. He wrote, "According to laboratory studies and limited experience, the nitrogen method should be effective and economical for commercial application." His recommendations--which may find interest from the citrus packer--are two:

1. Introducing nitrogen into the cans of a size larger than eight ounces by breaking the vacuum of

the sealing machine with nitrogen.

2. Purging the head space of the container with nitrogen before sealing.

Bayes of the same company published a few months later (5) an article recommending the removal of oxygen from liquid systems by means of counter-current gas stripping operations. He gave the following as the principal advantages of gas displacement methods over vacuum deaerating processes:

1. Minimum removal of desirable dissolved volatiles such as flavor essences and aroma.

2. Protection of the product after deaeration.

3. Exclusion of atmospheric contact at earlier stages during processing operations by counter-current recycling of effluent gases.

Heid and Beisel (15) mentioned lime juice among other citrus juices that could be concentrated by the new method which applies both sides of a refrigerant compressor system. By the time this is being written, it is reported that concentrated frozen lime juice is in production in Florida (22).

# CHAPTER III

## EXPERIMENTAL

### Source and Variety of Limes Used:

The limes used were provided by The Citrus Experiment Station, University of California, at Riverside. The information provided by Dr. W. P. Bitters of this Experiment Station states that they are of "the true limes, Citrus aurantifolia (Christm.) Swingle. They are known as the Mexican group, but are very frequently referred to also as the Key lime, or the West Indian lime." He adds, "Size is usually broken down into Pewees, Mediums, Jumbos and Giants. The size of such fruit varies from 1-3/8 inches to 1-7/8 inches." He continues to say, "its greatest overall disadvantage is the fruit is too small."

### Extracting the Juice:

A Sunkist electric juicer was used for extracting the juice after removing the metal parts to prevent the juice from being contaminated with the metal. The limes were first halved, using a stainless steel knife, then each half was pressed against the rotating porcelain burr until the juice was extracted. Sometimes the peel became cracked on account of the larger size of the burr,

causing some contamination with peel oil. Also, some of the seeds were crushed occasionally and might have yielded some of their oil. The juice was then strained through muslin.

In the preliminary work and one of the early batches, the limes were heated in a 2% calcium chloride solution at 185 - 190° F. for 15 to 20 minutes before juicing. The purpose of heating was to eliminate contamination with the peel oil (18) and the calcium chloride was thought to serve as a bactericidal agent. It was discovered that this treatment contributes an off-flavor to the juice. Samples of limeade were prepared from limes heated as usual, and from lime juice squeezed from limes without heating. They were submitted to tasters (20) and the acceptability scores were as follows, on basis of 10 = ideal and 4 = unacceptable.

Fresh heated limes: 3.85

Fresh unheated limes: 7.14

Nine tasters participated and seven detected the duplicate.

For purpose of comparison, it may be stated that the score of juice from limes stored about three weeks at 32° F. was 4.4 when heated before pressing. It is not known whether this higher score was due to storage or was simply a sample difference.

As a result of this taste test, the process of heating the limes before extracting the juice was discontinued.

Always glass containers were used, except for the stainless steel knife used for cutting the limes.

Containers:

Shelline polyethyline bags were used to contain the juice. Each one contained about 100 c.c. juice and each three bags receiving the same treatment were put unsealed in a No. 2 can so that vacuum sealing could be possible.

Freezing Scheme:

To test the three alternatives:

1. Pasteurization and non-pasteurization.

2. Sealing in a nitrogen atmosphere and sealing in a non-nitrogen atmosphere.

3. Vacuum sealing and ordinary sealing.

The following scheme was followed:

Pasteurization

| Treated with Nitrogen | | Non-treated with Nitrogen | |
|---|---|---|---|
| (1) Vacuum Sealed | (2) Sealed without Vacuum | (3) Vacuum Sealed | (4) Sealed without Vacuum |

Non-Pasteurization

| Treated with Nitrogen | | Non-treated with Nitrogen | |
|---|---|---|---|
| (5) Vacuum Sealed | (6) Sealed without Vacuum | (7) Vacuum Sealed | (8) Sealed without Vacuum |

There are then eight treatments.

Pasteurization was carried out by putting the bags containing the juice in boiling water for two minutes. Nitrogen was applied by passing it from a nitrogen cylinder through a pipette to the juice in the bottom of the bag for two minutes. The purpose was to displace the air in the juice and above the surface of the juice by nitrogen. When this was applied on the pasteurized samples, this was done at the same time as pasteurization. The pressure gauge on the nitrogen cylinder registered 5 lbs./square inch in both cases. About 25 inches of vacuum were used.

Freezing:

The cans were put in the quick freezer usually operating at 0° F. and, after freezing, they were transferred to the zero degree room.

Determination of Ascorbic acid:

The sodium salt of 2,6 dichlorophenol benzophenol was used for ascorbic acid determination. The

titration method was used on the first samples, and the photometric method was used on the frozen stored samples. It was reported by French and Abbott (14) that photometric, electrometric and titration methods for ascorbic acid determination all agreed when used on the same samples.

Determination of sugars:

The Munson-Walker method was used for the determination of reducing and total sugars.

Determination of Acidity:

A 1N sodium hydroxide solution was used for the determination of the acidity of the juice. Phenolphthalein was used as an indicator. Per cent acidity was calculated as citric acid according to the following formula: (26)

$$\frac{\text{Ml.NaOH X N}}{1000} \times \frac{\text{eq. wt. X 100}}{\text{sample weight}} = \% \text{ acidity}$$

Composition of the juice:

| | Ascorbic Acid | Acidity as Citric Acid | pH | Reducing Sugars | Sucrose | Total Sugars |
|---|---|---|---|---|---|---|
| | Mg./ 100 Ml. | % | | gr./ 100 Ml. | gr./ 100 Ml. | gr./ 100 Ml. |
| First Batch | 49.2 | 8.68 | 2.4 | 0.2374 | 0.2413 | 0.4787 |
| Second Batch | 36.02 | 7.42 | 2.8 | 0.4592 | 0.0627 | 0.5219 |

Determination of Desirable sugar: acid ratio:

A series of taste tests was carried out to determine the desirable sugar: acid ratio. Limeades having ratios of 11:1, 10:1, 9.4:1 and 8:1 were submitted simultaneously to 12 tasters and were scored as follows: (Ideal = 10, unacceptable = 4)

| Sugar:acid ratio | Score |
|---|---|
| 10:1 | 6.9 |
| 11:1 | 6.75 |
| 9.4:1 | 6.08 |
| 8.0:1 | 5.5 |

Following that, ratios of 12:1, 11:1 and 10:1 were submitted simultaneously to the tasters and they were given the following average scores:

| Sugar:acid ratio | Score |
|---|---|
| 12:1 | 7.0 |
| 11:1 | 6.9 |
| 10:1 | 6.01 |

Thirteen tasters participated in this taste test. It was decided to use the ratio of 12:1 in all the subsequent taste tests.

Determination of Suitable Dilution:

Limeade samples having a sugar acid ratio of 12:1 but prepared according to the following dilutions

were submitted to the tasters and were given the following scores:

| Dilution | Score |
|---|---|
| 1:7 | 7 |
| 1:9 | 5.9 |
| 1:11 | 4.3 |

So the dilution of 1:7 was always followed in all the following taste tests along with the sugar acid ratio of 12:1.

Testing the Frozen Samples:

Water added to the frozen juice to dilute it 1 to 7 thawed the samples in a comparatively short time. The juice frozen in each bag was thawed separately and then a sample for ascorbic acid determination was taken from the juice of each bag, thus making three replicates. For the taste test, the remaining juice from the three bags was combined to prepare a limeade having a sugar: acid ratio of 12:1. No special order was followed for taking the samples from the zero degree room, so they were taken at random.

As there were eight treatments and the limit is four samples per each sitting including a duplicate, three days were necessary to taste test the eight treatments. Three treatments were tested on each of the first

and second day and two on the third.

The procedure of the Food Technology Department, Oregon State College (20), was used for performing the taste tests and the evaluation of the significant scores. This procedure allows a difference between the scores assigned to the duplicates equivalent to 20% of the range between the highest and lowest scores assigned to the samples by the same person at this particular test.

The Taste Test Panels:

As the tasters form the panel--which is used as an instrument to measure the flavor--any invalidity or inaccuracy or failure to reproduce the same results on replicates will be reflected on the flavor scores. From Tables 2, 3, 6, and 7, we can see that the per cent of significant tasters neither improved nor was it very constant but it differed from one sitting to the other, sometimes for the better, at other times for the worse. In an attempt to investigate validity, accuracy and reliability of the taste panel, a series of limeade taste tests were carried out. This was, also, hoped to train the tasters to taste limeade discerningly.

Table 2

Panel Performance on Canned, Fresh and Blended Limeades

(Average scores of judges who detected duplicates)

| | First Day | | Second Day | | Third Day | |
|---|---|---|---|---|---|---|
| | A.M. | P.M. | A.M. | P.M. | A.M. | P.M. |
| Canned Limeade | 7 & 7 | | 5.66 | 6.75 | 7.1 & 7.1 | 5.1 |
| Fresh Limeade | 6.9 | 7.9 & 7.9 | 6.33 & 6.33 | 5.25 & 5.25 | 6.3 | 7 & 7 |
| A 50/50 blend | | 6.6 | 6.66 | 6.5 | 7 | 6.6 |
| No. of Tasters | 10 | 8 | 6 | 4 | 10 | 9 |
| Per cent of significant tasters | 90 | 87.5 | 50 | 57.1 | 70 | 77.8 |

Ideal = 10 Not Acceptable = 4

Table 3

Flavor Scores of February Batch Openned After 6.5 Months Showing Reliability of Taste Panel

| Treatment | Date Op'd | 11 A.M. No. of Tasters | 11 A.M. % Significant Tasters | 11 A.M. Significant Score | 4 P.M. No. of Tasters | 4 P.M. % Significant Tasters | 4 P.M. Significant Score |
|---|---|---|---|---|---|---|---|
| (1) Pasteurized $N_2$ Vacuum Sealed | 8/21 1950 | 9 | 55.6 | 6.2 | 8 | 25 | 7.5& 7.5 |
| (2) Pasteurized $N_2$ Sealed without Vacuum | 8/21 1950 | 9 | 55.6 | 6.2 | 8 | 25 | 7.5 |
| (3) Pasteurized No$N_2$ Vacuum Sealed | 8/22 1950 | 8 | 37.5 | 5.66 | 7 | 57.1 | 7.25& 7.25 |
| (4) Pasteurized No$N_2$ Sealed without Vacuum | 8/23 1950 | 9 | 55.6 | 7.6 | 10 | 40 | 7 & 7 |
| (5) Non-Pasteurized $N_2$ Vacuum Sealed | 8/21 1950 | 9 | 55.6 | 7.2& 7.2 | 8 | 25 | 7 |

(Continued on next page)

Table 3 (Continued)

| Treatment | Date Op'd | 11 A.M. No. of Tasters | 11 A.M. Significant Tasters | 11 A.M. Significant Score | 4 P.M. No. of Tasters | 4 P.M. Significant Tasters | 4 P.M. Significant Score |
|---|---|---|---|---|---|---|---|
| (6) Non-Pasteurized $N_2$ Sealed without Vacuum | 8/23 1950 | 9 | 55.6 | 7.2& 7.2 | 10 | 40 | 7.75 |
| (7) Non-Pasteurized No$N_2$ Vacuum Sealed | 8/22 1950 | 8 | 37.5 | 5.66 | 7 | 57.1 | 7.5 |
| (8) Non-Pasteurized No$N_2$ Sealed without Vacuum | 8/22 1950 | 8 | 37.5 | 6.66& 6.66 | 7 | 57.1 | 7.75 |
| Mean | | | | 6.67 | | | 7.41 |

Ideal = 10 Not Acceptable = 4

Samples of limeade consisted of:

1. Fresh limeade using limes obtained from the market.

2. Canned limeade secured from the market as such.

3. A 50/50 blend of the fresh and the canned limeades.

The number of judges varied from four to ten and the panels consisted of the same persons largely but not entirely. The results of the taste tests which were carried out on three consecutive days, morning and afternoon, appear in Table 2. An examination of these results show:

1. The tasters did not only twist from preferring one sample to the other on different days but also on the same day from morning to afternoon, and, surprisingly, the fresh limeade was frequently given the lowest score.

2. The per cent of significant judges was not consistent but differed from one sitting to the other.

3. The scores assigned to the different samples had a fairly narrow range, from 5.14 to 7.86.

The samples of the February batch openned after six and one-half months storage also were submitted to the tasters twice--one at 11 A.M., the other at 4 P.M. Table 3 shows how differently they were scored from morning to afternoon. Besides, it shows the inconsistency

of per cent significant judges. Even the mean of the scores assigned to the different samples in the morning differ from that assigned to the same samples in the afternoon, being 6.67 in the morning and 7.41 in the afternoon.

Assigning different scores for the same sample in the morning and afternoon was done by tasters who were correct in detecting the duplicates as much as 70% of the time. Personal interviews conducted by the writer with some tasters immediately after they were through testing and before revealing the identity of the samples gave not only different but sometimes opposite explanations for the preference of the samples after the different sittings.

Summary on Panel Performance:

An over-all examination of the Flavor Score tables shows some irregularity. No one reason could be justifiably and exclusively assigned. However, one or more of the following factors could contribute to this irregularity:

1. The panel of tasters was inexperienced on lime juice and, consequently, rather non-discriminating as was discussed previously.

2. The difference between the samples could

be too small for the tasters to detect.

3. Taste is very much affected by the environment such as temperature and the condition of the taster himself. The temperature of the room varied considerably due to the weather.

4. Performing the taste tests on three consecutive but different days was shown to lead to considerable inconsistency on part of the judges.

# CHAPTER IV

## DISCUSSION AND RESULTS

It was mentioned before that ascorbic acid determination and organolyptic flavor tests were carried out. Close observation showed no change in color in any case. The frozen juice always kept its yellowish green natural color. No turbidity or any other related changes were observed. It is regretted that the design of the experiment did not allow statistical evaluation of the results, yet the writer will discuss the results in the following pages beginning with ascorbic acid and then flavor tests.

### I. Ascorbic acid content

The results of ascorbic acid retention are shown in Figures 1 to 4 while Tables 4 and 5 are analyses of ascorbic acid retention after different storage periods to enable comparison of the effect of different variables on the vitamin retention. Table 4 is for the January batch and Table 5 for the February batch.

1. Pasteurization and Applying Nitrogen:

a. Pasteurization in an atmosphere of nitrogen versus pasteurization without applying nitrogen (in air):

By examining the figures in Tables 4 and 5, it

Table 4

Analysis of Ascorbic Acid Content of 1st Batch
Frozen January 9, 10 and 11, 1950

| | Pasteurized | | | | | | | | Non Pasteurized | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Nitrogen | | | | No Nitrogen | | | | Nitrogen | | | | No Nitrogen | | | | | |
| | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | Totals | Means |
| | Mg./100 Ml. | | | | | | | | Mg./100 Ml. | | | | | | | | | |
| **Vacuum:** | | | | | | | | | | | | | | | | | | |
| after 3 mos. | 28 | 31 | 22 | 27 | 22 | 28 | 22 | 24 | 25 | 25 | 24 | 25 | 25 | 21 | 28 | 25 | 101 | 25.25 |
| after 4.5 mos. | 27 | 25 | 26 | 26 | 23 | 25 | 24 | 24 | 25 | 25 | 25 | 25 | 25 | 24 | 26 | 25 | 100 | 25.00 |
| after 6.5 mos. | 27.3 | 27 | 28 | 27.3 | 25 | 24 | 27 | 25.3 | 26 | 23.8 | | 24.9 | 23.8 | 27 | 26 | 25.6 | 103.1 | 25.78 |
| **No Vacuum:** | | | | | | | | | | | | | | | | | | |
| after 3 mos. | 28 | 22 | 31 | 27 | 22 | 22 | 25 | 23 | 28 | 22 | 25 | 25 | 25 | 31 | 31 | 29 | 104 | 26.0 |
| after 4.5 mos. | 28 | 26 | 27 | 27 | 22 | 23 | 22 | 22.3 | 26 | 23 | 25 | 24.7 | 28 | 29 | 29 | 28.66 | 102.62 | 25.65 |
| after 6.5 mos. | 19.6 | 28 | 24.5 | 24 | 23.8 | 23.8 | 22 | 23.2 | 22.4 | 21 | 24.5 | 22.6 | 27.3 | 27 | 23.8 | 26 | 95.8 | 23.95 |
| **Totals:** | | | | | | | | | | | | | | | | | | |
| after 3 mos. | | | | 54 | | | | 47 | | | | 50 | | | | 54 | | |
| after 4.5 mos. | | | | 53 | | | | 46.3 | | | | 49.66 | | | | 53.66 | | |
| after 6.5 mos. | | | | 51.3 | | | | 48.5 | | | | 47.5 | | | | 51.6 | | |
| **Means:** | | | | | | | | | | | | | | | | | | |
| after 3 mos. | | | | 27 | | | | 23.5 | | | | 25 | | | | 27 | | |
| after 4.5 mos. | | | | 26.5 | | | | 23.15 | | | | 24.83 | | | | 26.83 | | |
| after 6.5 mos. | | | | 25.65 | | | | 24.25 | | | | 23.75 | | | | 25.8 | | |

Table 5

Analysis of Ascorbic Acid Content of 2nd Batch
Frozen February 6 and 7, 1950

| | Pasteurized | | | | | | | | Non Pasteurized | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Nitrogen | | | | No Nitrogen | | | | Nitrogen | | | | No Nitrogen | | | | | |
| | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | 1st bag | 2nd bag | 3rd bag | Ave. | Totals | Means |
| | Mg./100 Ml. | | | | | | | | Mg./100 Ml. | | | | | | | | | |
| Vacuum: | | | | | | | | | | | | | | | | | | |
| after 3 mos. | 30 | 31.5 | 33 | 31.5 | 29 | 27 | 32.9 | 29.6 | 33.6 | 28 | 33.6 | 31.7 | 28 | 29.4 | 28.7 | 28.7 | 121.5 | 30.4 |
| after 6.5 mos. | 27 | 23.8 | 27.3 | 26 | 21 | 30.8 | 23.8 | 25.2 | 24.5 | 28 | 24.5 | 25.66 | 22.5 | 30.4 | 28 | 26.97 | 103.83 | 25.96 |
| Non Vacuum: | | | | | | | | | | | | | | | | | | |
| after 3 mos. | 28.5 | 31 | 32 | 31 | 30 | 28 | 32.9 | 30.3 | 33.6 | 28 | | 30.6 | 30 | 32 | 31 | 31 | 123.1 | 30.8 |
| after 6.5 mos. | 24.5 | 29.4 | 24 | 26 | 22.5 | 22.5 | 28 | 24.33 | 28 | 23.8 | 29.4 | 27.66 | 22.5 | 27 | 23.8 | 24.43 | 97.26 | 24.32 |
| Totals: | | | | | | | | | | | | | | | | | | |
| after 3 mos. | | | | 62.5 | | | | 59.9 | | | | 62.3 | | | | 59.7 | | |
| after 6.5 mos. | | | | 52 | | | | 49.53 | | | | 53.32 | | | | 53.39 | | |
| Means: | | | | | | | | | | | | | | | | | | |
| after 3 mos. | | | | 31.25 | | | | 29.95 | | | | 31.15 | | | | 29.85 | | |
| after 6.5 mos. | | | | 26 | | | | 24.77 | | | | 26.66 | | | | 26.7 | | |

was noticed that samples pasteurized in a nitrogen atmosphere always showed a higher content of ascorbic acid than those of the samples pasteurized without applying nitrogen (in air).

These results should be expected because heating is more destructive to ascorbic acid in an atmosphere of air than in an inert atmosphere such as that of nitrogen.

b. Pasteurization in atmosphere of Nitrogen versus Non-Pasteurization in atmosphere of Nitrogen:

The ascorbic acid content of the pasteurized samples with nitrogen was higher than the non-pasteurized samples with nitrogen, except in two cases both in the February batch, wherein there are minor irregularities.

The differences of ascorbic acid content between both treatments are small--the highest being 2.4 mg. ascorbic acid per 100 c.c. juice. These small differences are insignificant and cannot be counted on.

c. Pasteurization without Applying Nitrogen versus Non-Pasteurization without Applying Nitrogen:

The non-pasteurized no-nitrogen samples show higher ascorbic acid content than the pasteurized no-nitrogen samples. The highest difference is 6 mg. per 100 c.c. juice in the samples sealed without vacuum after three months storage of the January batch.

The only exceptions are the samples vacuum sealed in the February batch after three months storage where the pasteurized no-nitrogen sample showed a content of ascorbic acid corresponding to 0.9 mg. per 100 c.c. of the juice higher than the ascorbic acid content of the non-pasteurized no-nitrogen sample.

The higher ascorbic acid content of the non-pasteurized samples are attributed to the loss of the vitamin during pasteurization in the presence of the atmospheric oxygen.

2. Vacuum Sealing versus Non-Vacuum Sealing:

The means of the ascorbic acid content of vacuum sealed samples are slightly lower than those of the samples sealed without vacuum after three months and four and one-half months, but higher after six and one-half months. It is possible that the beneficial effect of vacuum sealing appears in the longer storage periods. The lower content of ascorbic acid in the non-vacuum sealed samples after a storage period of six and one-half months could be explained to be due to the slow rate of ascorbic acid oxidation through the effect of oxygen present in the non-vacuum sealed cans, at the temperature used in storage ($0^{o}$ F.)

3. Effect of Storage Period:

A look on the graphs in Figures 1, 2, 3, and 4

shows a distinct drop in ascorbic acid content from 49.2 mg. original ascorbic acid content/100 c.c. juice to a content of the neighborhood of 25 mg. per 100 c.c. juice after three months storage in the January batch. In the February batch, the respective drop is from an original ascorbic acid content of 36.02 mg. per 100 c.c. juice to a content of about 31 mg. per 100 c.c. juice. The losses--if any-- after longer storage periods are comparatively slight in both batches.

The losses which appear on the graphs in the first three months storage occurred largely during preparation, as January batches took about eight hours from time of extracting the juice until the cans were put in the sharp freezer, while February batches took slightly over five hours for preparation.

As the batches which were openned after different periods of storage were prepared and frozen on subsequent but different days, it is not felt justifiable to use their figures as if they were for one sample stored for different periods of time. Their preparation on different days could explain the irregularities in the graphs. (Compare graphs in Figures 1, 2, 3, and 4.)

II. Flavor Scores

Taste tests were carried out to find out the

effect of preparation methods on the flavor of the juice, by preparing limeade samples from the frozen juice always having a sugar:acid ratio of 12:1 and dilution of 1:7. The previously mentioned Food Technology Department procedure (20) was used in this evaluation. Tables 6 and 7 show the assigned scores, while Tables 8 and 9 are an analysis of these scores. The writer will discuss the taste tests, the scores and their analysis as shown in Tables 8 and 9 in the following pages.

1. Pasteurization and Applying Nitrogen:

a. Pasteurization in a Nitrogen atmosphere versus Pasteurization without Applying Nitrogen:

Samples pasteurized in the absence of nitrogen always scored higher than those pasteurized in the presence of nitrogen except in two cases. In the January batch after four and one-half months, the pasteurized nitrogen vacuum sealed sample scored 7 while its corresponding sample without nitrogen scored 6 only. In the February batch after six and one-half months, the vacuum samples--nitrogen treated and non-nitrogen treated--scored the same 6.6.

Those lower scores for the nitrogen treated samples could be due to the loss of some of the volatile substances responsible for flavor carried with the stream of nitrogen which was applied during pasteurization.

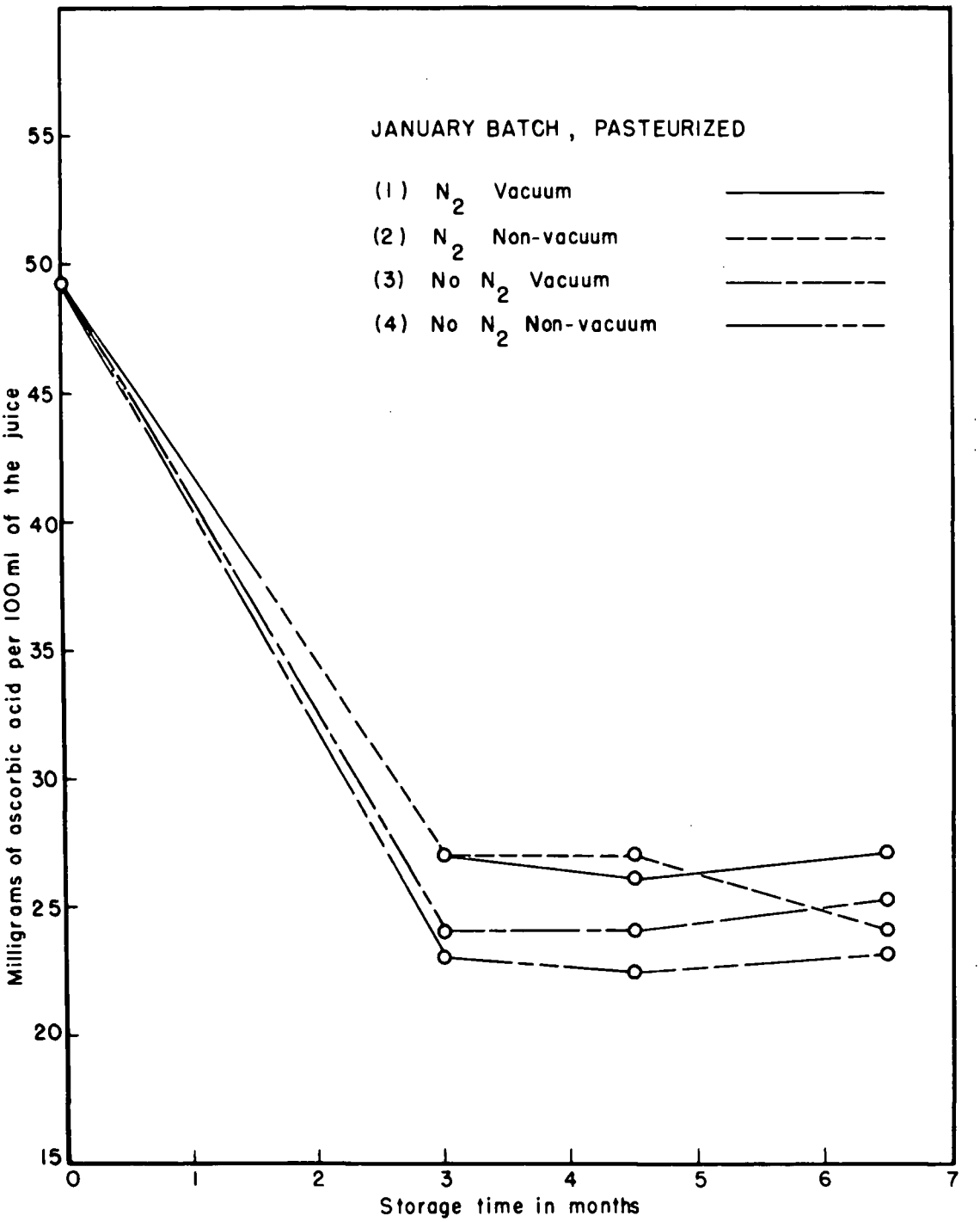


**Figure 1. Ascorbic acid content of stored frozen lime juice; January batch, pasteurized.**

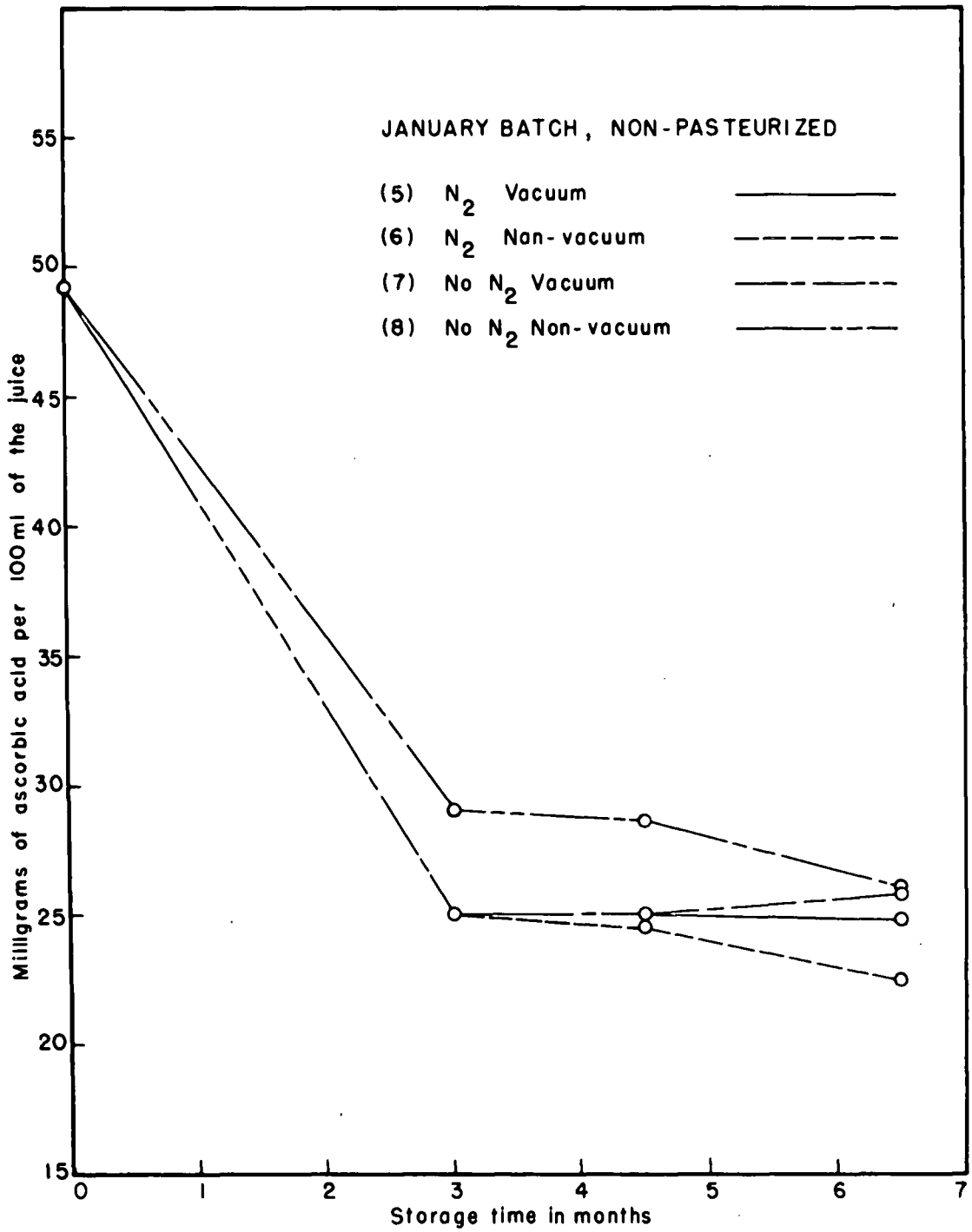


**Figure 2. Ascorbic acid content of stored frozen lime juice; January batch, non-pasteurized.**

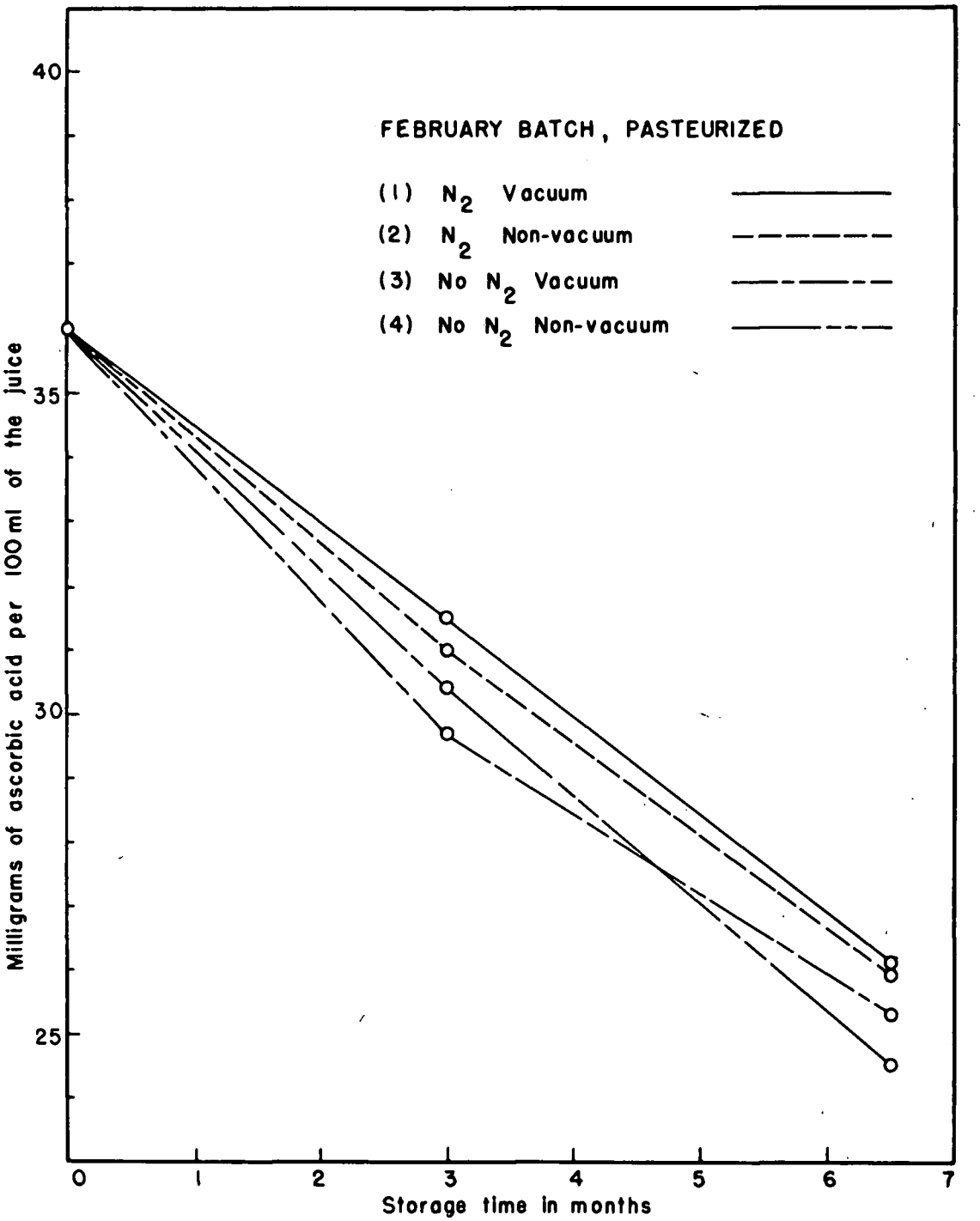


Figure 3. Ascorbic acid content of stored frozen lime juice; February batch, pasteurized.

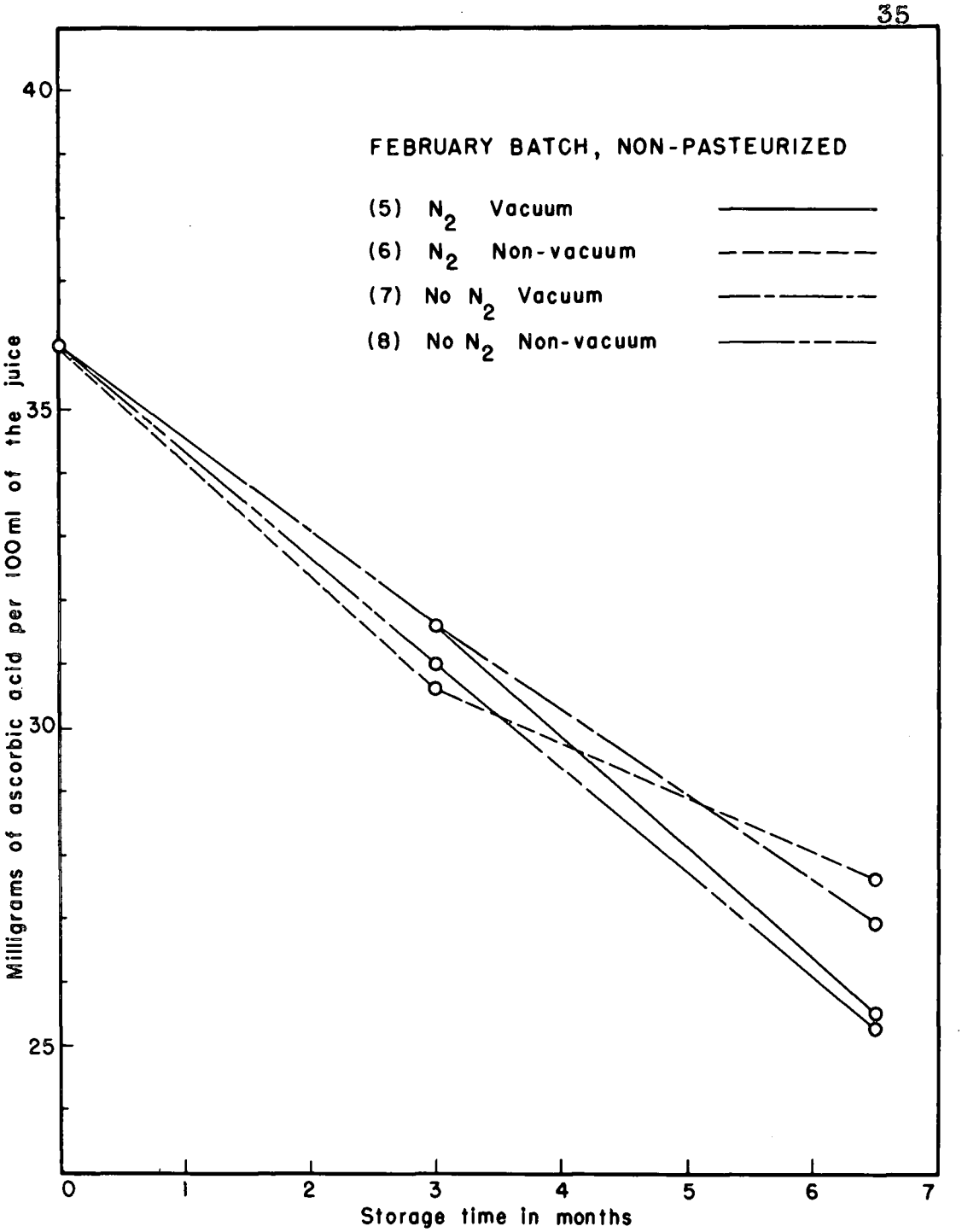


**Figure 4. Ascorbic acid content of stored frozen lime juice; February batch, non-pasteurized.**

Table 6

Flavor Scores of First Batch
Frozen January 9, 10 and 11, 1950

| Treatment | After 3 Months Storage | | | | After 4.5 Months Storage | | | | After 6.5 Months Storage | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Jan. 11 | | Apr.12-14'50 | | Jan. 9 | | May 22-24'50 | | Jan. 10 | | July 26-28'50 | |
| | No. of Tasters | % Significant Tasters | Significant Score | Total Score | No. of Tasters | % Significant Tasters | Significant Score | Total Score | No. of Tasters | % Significant Tasters | Significant Seore | Total Score |
| (1) Pasteurized $N_2$ Vacuum Sealed | 14 | 64.3 | 5<br>7& | 5.2<br>6.2& | 13 | 23.1 | 7<br>7& | 6.9<br>6.2& | 11 | 54.6 | 6.8 | 6.8 |
| (2) Pasteurized $N_2$ Sealed without Vacuum | 14 | 35.7 | 6 | 6.1 | 13 | 23.1 | 5.66<br>6& | 6.5<br>6.4& | 11 | 54.6 | 7 | 6.8 |
| (3) Pasteurized No $N_2$ Vacuum Sealed | 11 | 9.1 | 8<br>7& | 6.6<br>6.4& | 8 | 75 | 6 | 6.1 | 10 | 10 | 8 | 7.3 |
| (4) Pasteurized No $N_2$ Sealed without Vacuum | 14 | 64.3 | 7<br>7& | 6.6<br>6.5& | 8 | 75 | 6.33 | 6.6 | 11 | 54.6 | 7.5 | 7.5 |
| (5) Non Pasteurized $N_2$ Vacuum Sealed | 11 | 9.1 | 7 | 6.6 | 8 | 75 | 6.33 | 6.4 | 10 | 10 | 8<br>8& | 6.9<br>7& |
| (6) Non Pasteurized $N_2$ Sealed without Vacuum | 14 | 35.7 | 7.2<br>6.4& | 6.9<br>6.2& | 11 | 18.2 | 5.5<br>6& | 6.8<br>5.9& | 10 | 10 | 8<br>7.5& | 7.5<br>7.4& |
| (7) Non Pasteurized No $N_2$ Vacuum Sealed | 14 | 35.7 | 6.4 | 6.5 | 11 | 18.2 | 6 | 6.5 | 11 | 54.6 | 7.5<br>7& | 7.3<br>6.8& |
| (8) Non Pasteurized No $N_2$ Sealed without Vacuum | 14 | 64.3 | 6.66 | 6.9 | 11 | 18.2 | 6.5 | 6.4 | 11 | 54.6 | 7 | 6.6 |

Ideal - 10

Not Acceptable - 4

Table 7

Flavor Scores of Second Batch
Frozen February 6th and 7th, 1950

| | After 3 Months Storage | | | | After 6.5 Months Storage * | | | |
|---|---|---|---|---|---|---|---|---|
| | Feb. 7 | | May 10-12, '50 | | Feb. 6 | | Aug. 22-24, '50 | |
| | No. of Tasters | % Signi-ficant Tasters | Signi-ficant Score | Total Score | No. of Tasters | % Signi-ficant Tasters | Signi-ficant Score | Total Score |
| (1) Pasteurized $N_2$ Vacuum Sealed | 12 | 66.6 | 5.8 | 5.5 | 17 | 41.2 | 6.8 | 6.6 |
| (2) Pasteurized $N_2$ Sealed without Vacuum | 12 | 66.6 | 5.75 | 5.25 | 17 | 41.2 | 6.8 | 6.4 |
| (3) Pasteurized No $N_2$ Vacuum Sealed | 10 | 70 | 6.4 | 6.6 | 15 | 46.7 | 6.6 | 6.7 |
| (4) Pasteurized No $N_2$ Sealed without Vacuum | 10 | 70 | 7& 7 | 6.9& 6.9 | 19 | 47.4 | 6.8 | 7.2 |
| (5) Non Pasteurized $N_2$ Vacuum Sealed | 12 | 25 | 6.66& 6.66 | 6.6& 6.75 | 17 | 41.2 | 7.1 | 7 |
| (6) Non Pasteurized $N_2$ Sealed without Vacuum | 12 | 25 | 6.66 | 6.5 | 19 | 47.4 | 7.4 | 7 |
| (7) Non Pasteurized No $N_2$ Vacuum Sealed | 10 | 70 | 6.9 | 6.9 | 15 | 46.7 | 6.7 | 6.7 |
| (8) Non Pasteurized No $N_2$ Sealed without Vacuum | 12 | 66.6 | 7.1& 7.1 | 7.1& 6.33 | 15 | 46.7 | 7.3 | 6.8 |

* This batch was submitted twice to the tasters, one at 11 A.M., the other at 4 P.M. The scores reported here are the weighed ones of both panels.

Ideal - 10

Not Acceptable - 4

Table 8

Analysis of Flavor Scores* of 1st Batch
Frozen January 9, 10 and 11, 1950

| | Pasteurized | | Non-Pasteurized | | | |
|---|---|---|---|---|---|---|
| | Nitrogen | No Nitrogen | Nitrogen | No Nitrogen | Totals | Means |
| Vacuum: | | | | | | |
| after 3 mos. | 5 | 8 | 7 | 6.4 | 26.4 | 6.6 |
| after 4.5 mos. | 7 | 6 | 6.33 | 6 | 25.33 | 6.33 |
| after 6.5 mos. | 6.8 | 8 | 8 | 7.5 | 30.3 | 7.6 |
| Non-Vacuum: | | | | | | |
| after 3 mos. | 6 | 7 | 7.2 | 6.66 | 26.86 | 6.7 |
| after 4.5 mos. | 5.66 | 6.33 | 5.5 | 6.5 | 23.99 | 6 |
| after 6.5 mos. | 7 | 7.5 | 8 | 7 | 29.5 | 7.4 |
| Totals: | | | | | | |
| after 3 mos. | 11 | 15 | 14.2 | 13.56 | | |
| after 4.5 mos. | 12.66 | 12.33 | 11.83 | 12.5 | | |
| after 6.5 mos. | 13.8 | 15.5 | 16 | 14.5 | | |
| Means: | | | | | | |
| after 3 mos. | 5.5 | 7.5 | 7.1 | 6.5 | | |
| after 4.5 mos. | 6.33 | 6.2 | 5.9 | 6.25 | | |
| after 6.5 mos. | 6.9 | 7.75 | 8 | 7.25 | | |

* Scores used here are the significant scores.

Ideal - 10 Not Acceptable - 4

Table 9

Analysis of Flavor Scores* of 2nd Batch
Frozen February 6 and 7, 1950

| | Pasteurized | | Non-Pasteurized | | | |
|---|---|---|---|---|---|---|
| | Nitrogen | No Nitrogen | Nitrogen | No Nitrogen | Totals | Means |
| Vacuum: | | | | | | |
| after 3 mos. | 5.8 | 6.4 | 6.66 | 6.9 | 25.76 | 6.4 |
| after 6.5 mos. | 6.6 | 6.6 | 7.1 | 6.7 | 27 | 6.75 |
| Non-Vacuum: | | | | | | |
| after 3 mos. | 5.75 | 7 | 6.66 | 7.1 | 26.51 | 6.6 |
| after 6.5 mos. | 6.6 | 6.8 | 7.4 | 7.3 | 28.1 | 7 |
| Totals: | | | | | | |
| after 3 mos. | 11.55 | 13.4 | 13.32 | 14 | | |
| after 6.5 mos. | 13.2 | 13.4 | 14.5 | 14 | | |
| Means: | | | | | | |
| after 3 mos. | 5.8 | 6.7 | 6.66 | 7 | | |
| after 6.5 mos. | 6.6 | 6.7 | 7.25 | 7 | | |

*Scores used here are the significant scores.

Ideal = 10 Not Acceptable = 4

b. Pasteurization in an atmosphere of Nitrogen versus Non Pasteurization in an atmosphere of Nitrogen:

Non-pasteurized nitrogen treated samples always scored higher than pasteurized nitrogen treated samples except in two cases. Both cases are in the January batch openned after four and one-half months storage.

These higher scores of the non-pasteurized samples show the undesirable effect of pasteurization on the flavor of lime juice even in the absence of oxygen.

c. Pasteurization versus Non Pasteurization:

Averages of the means of the scores of the pasteurized and non-pasteurized samples are as follows:

| | Pasteurized | Non-Pasteurized |
|---|---|---|
| January batch | 6.7 | 6.8 |
| February batch | 6.4 | 7.0 |

d. Application of Nitrogen versus Non Application of Nitrogen:

Following are the averages of the scores of the nitrogen treated samples and those not treated with nitrogen:

| | Nitrogen treated | No Nitrogen treatment |
|---|---|---|
| January batch | 6.6 | 6.9 |
| February batch | 6.6 | 6.9 |

The differences could be explained to be due to the loss of some flavor elements during application of nitrogen or it could be due to an undesirable effect of nitrogen impurities on the flavor of the juice.

2. Vacuum versus Non Vacuum:

Tables 6 and 7 show that vacuum sealed samples scored lower than the samples sealed without vacuum after three months storage, but they were assigned higher scores after four and one-half months and after six and one-half months storage. This shows that the vacuum desirable effect appears in the longer storage periods. It is in agreement with what was found in the effect of vacuum sealing on the retention of ascorbic acid.

A look through the averages of the significant scores and those of the total scores in Tables 6 and 7 let the observer notice that they do not differ greatly, the greatest difference being 1.4 in the pasteurized no nitrogen vacuum sealed sample after three months storage of the January batch. One also can see that the significant and the total scores assigned for a particular sample are the same in several cases. Since it is also apparent from the same tables that except for one sample which scored 5 = acceptable, the scores ranged between 6 = fairly good and 8 = very good, we can say

that the tasters liked the product whether they were able to detect the duplicate or not.

# CHAPTER V

## SUMMARY AND CONCLUSIONS

Lime juice from Mexican (true) limes was frozen in this investigation. Polyethethylene bags were used as containers for the juice. The bags were put in No. 2 cans and the cans sealed. Eight treatments representing the variables, pasteurization, applying nitrogen, vacuum sealing and their opposites were investigated. Ascorbic acid was determined in the fresh samples by the titration method using the sodium salt of 2-6 dichlorophenol benzo-phenol dye, while it was determined in the frozen thawed samples, using the same compound, by the colorimetric method using an electrophotometer. Taste tests by means of panels consisting of 14 to 7 judges were carried out to evaluate the flavor of the frozen product.

The conclusions are as follows:

1. Pasteurization in an atmosphere of nitrogen protects the ascorbic acid, but the flavor scores assigned to the nitrogen treated pasteurized samples were lower than those assigned to the samples pasteurized without applying nitrogen.

2. While differences in ascorbic acid content between samples pasteurized in nitrogen atmosphere and those non pasteurized nitrogen treated samples are small,

the non pasteurized nitrogen treated samples were assigned higher flavor scores. This shows the undesirable effect of pasteurization on the flavor of lime juice even in the absence of oxygen.

3. The non pasteurized no nitrogen samples had higher ascorbic acid contents than the samples Pasteurized without applying Nitrogen. This appears to be due to the destruction of ascorbic acid during pasteurization in the presence of the atmospheric oxygen. Flavor scores assigned to these samples were inconsistent.

4. Averages of flavor scores of the non pasteurized samples were slightly higher than those of the pasteurized samples. The differences in the respective figures for ascorbic acid are very small.

5. Averages of flavor scores of no nitrogen treated samples are higher than those of the nitrogen treated samples. Ascorbic acid contents of nitrogen treated samples are in most cases slightly higher than those of the non nitrogen treated samples.

6. Ascorbic acid contents and flavor scores of the non vacuum sealed samples are higher than those of the vacuum sealed samples after the short storage period but lower after the longer storage period. However, the differences are slight.

7. Most of the loss of ascorbic acid was

during preparation, while the loss during storage was small.

8. The process of freezing lime juice, even untreated, is successful as far as ascorbic acid retention and flavor are concerned. However, with other varieties vacuum sealing may be found to be worth the added expense, and treatment with recirculated nitrogen, not attempted here, may be recommended for further experimentation.

# BIBLIOGRAPHY


1. Anon. Frozen concentrated juice process contributes new know-how. Food Industries, 21:67-70, 92-95. July 1949.

2. Aref, Hussein. The Science of Agricultural Industries. (In Arabic). 2nd ed. Cairo, Egypt, 1946. Ietemad Press, 1015pp.

3. Banerjee, B. N. and Rang Rao, N. K. Utilization of oranges and limes. Agriculture and Livestock in India 6:139-153, 1936.

4. Bassili, Botross. General Director of the Department of Economics, Egyptian Ministry of Agriculture. Personal communication. August 11, 1950.

5. Bayes, A. L. Investigation on the use of nitrogen for the stabilization of perishable products. Food Technology 4:151-157. April 1950.

6. Burton, L. V. Florida produces frozen orange juice. Food Industries 3:208-211, 1931.

7. Camp, A. F. Proceedings of Florida State Horticultural Society, pp.60-65, 1932.

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

9. Chace, E. M. and Poore, H. D. Quick freezing citrus fruit juices and other fruit products. Industrial and Engineering Chemistry 23:1109-1112, 1931.

10. Cruess, W. V. Commercial Fruit and Vegetable Products. 3rd ed. New York, McGraw-Hill Book Company, Inc. 1948. 906pp.

11. Cruess, W. V. and Irish, J. H. Fruit beverage investigations, California Agriculture Experiment Station, p.359. April 1923.

12. El-Abd, A. The Utilization and Preservation of Limes. Master of Science Thesis. University of Massachusetts. 1942.

13. Finnegan, Wm. J. Preserving citrus juices by freezing. Ice and Refrigeration 88:51-54, 1935.

14. French, R. B. and Abbott, V. D. Levels of carotene and Vitamin C in Florida-grown foods. Florida Agriculture Experiment Station. Technical Bulletin 444, 1948.

15. Heid, J. L. and Beisel, C. G. Improved citrus concentrate depends on advanced technique. Food Industries 20:516-519. April 1948.

16. Joseph, G. H. Citrus products--a quarter century of amazing progress. Economic Botany 1:415-426, 1947.

17. Joslyn, M. A. The problem of preserving orange juice by freezing. Industrial and Engineering Chemistry 24:665-668, 1932.

18. Joslyn, M. A. and Marsh, G. L. Possibilities and limitations in canning orange juice. Food Industries 5:172-173, 1933.

19. Joslyn, M. A. and Marsh, G. L. Keeping quality of frozen orange juice. Industrial and Engineering Chemistry 26:295-299, 1934.

20. Lorant, G. and Wiegand, E. H. Testing acceptability of food products. Taste test procedure. Agriculture Experiment Station, Oregon State College. Circular of Information No. 246. January 1949.

21. Miller, E. V. The physiology of citrus fruits in storage. Citrus Industries 27, 4, 6-8, 20, 22. 1946.

22. Moore, E. L. Florida Citrus Commission, Lake Alfred, Florida. Personal communication. September 27, 1950.

23. Moursi, A. A. Agriculture Attache, Royal Egyptian Embassy, Washington, D. C., U.S.A. Personal communication. July 21, 1950.

24. Rosberg, N. H. Processing and handling of quick frozen orange juice. Refrigeration Engineering 35:19-22, 51. 1938.

25. Sedky, A., Fellers, C. R. and Esselen, W. B. Jr. An improved orange juice concentrate. Fruit Products Journal 21:5, 136-138. 1942.

26. Sivetz, M. Acids play important roles in flavor. Food Industries 21:10, 1384-1385. October 1949.

27. Stahl, A. L. Preservation of citrus juices and pulps. Florida Agriculture Experiment Station Annual Report p.109-110, 1939.

28. Stahl, A. L. Concentration of citrus juices by freezing. Citrus Industries 25:9, 5, 12. 1944.

29. Tempany, M. A. The preservation of citrus fruit juices. Imperial Institute Bulletin 36:334-349, 1938.

30. Tressler, D. K., Joslyn, M. A. and Marsh, G. L. Fruit and Vegetable Juices. New York. The AVI Publishing Company, Inc. 1939, 549pp.

31. Tressler, D. K. and Evers, C. F. The Freezing Preservation of Foods. New York. The AVI Publishing Company, Inc. 1947, 932pp.

32. Walker, W. S. How nitrogen protects the quality of foods. Food Industries 21:9, 1189-1190, 1342, 1344. September 1949.

33. Webber, H. J. and Batchelor, L. D. The Citrus Industry. V. I. Berkeley and Los Angeles. University of California Press, 1943. 1028 pp.

34. Wharton, M. New method of citrus juice packing. Ice and Refrigeration 92:281-283, 1937.