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EFFECT OF PRESERVATION METHODS AND STORAGE CONDITIONS ON
THE QUALITY OF THE JUICE FROM THREE SWEET ORANGE VARIETIES

by

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PRESERVATION OF ORANGE JUICE

Jafri

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ABSTRACT

Preservation of orange juice offers one way to utilize oranges during the peak season and to provide a constant supply of oranges in the form of juice throughout the year.

Experiment 1

Three important commercial varieties of sweet oranges (Bizzri, Shammouti and Valencia) grown in Lebanon were selected for the study. Juice from these varieties was preserved by pasteurization or by addition of sodium benzoate, sodium metabisulphite or sugar. The bottled juice was stored in plain, amber or green bottles on open or closed shelves for 12 months at room temperature. Observations on the ascorbic acid content, percentage of total soluble solids and total acids, pH, and taste were made initially and at the end of 3, 6, 9, and 12 months of storage.

Under all treatments and storage conditions, the rate of loss of ascorbic acid was faster during the first

3 months than during subsequent storage periods. There was a general increase in T.S.S. content, total acids and pH of the juice concurrent with the increase in the length of storage period. Furthermore, the taste deteriorated as the length of storage period increased.

Considering the varieties and treatments studied, Valencia orange juice preserved with sodium metabisulphite and stored on closed shelves in plain or amber bottles retained the highest content of ascorbic acid. The total soluble solids, total acid content and the pH of Valencia juice thus-treated were also higher as compared to the other varieties. The bisulphite-treated Valencia juice retained the best taste throughout the entire period of storage.

Experiment 2

A second experiment was carried out to study the effect of headspace on the retention of ascorbic acid. In this experiment, Valencia juice was preserved with sodium metabisulphite and stored in plain bottles, with or without headspace, in the open at room temperature. The juice was stored for a period of 15 weeks and ascorbic acid determinations were made initially and at the end of every third week. The results obtained show that the juice

bottled without headspace retained a higher percentage of ascorbic acid. Furthermore, the rate of loss of ascorbic acid was slower in the juice bottled without headspace as compared to the juice kept in bottles with a headspace of $2\frac{1}{2}$ inches.

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INTRODUCTION

Citrus is the most important fruit crop consumed as human food. In 1956-57 for example the world crop approached 18 million tons and was second only to grapes.¹

The demand for and acceptance of citrus fruits and, in particular, sweet oranges (Citrus sinensis) in the daily diet of humans are based largely on the nutritional value, taste, flavor and other aesthetic characteristics of the fruit such as color and texture. These quality factors are dependent directly on the structure and chemical composition of the fruit. Sweet oranges are a very good source of dietary vitamin C and, in addition, supplementary nutritional value is obtained from the amino acids, inorganic salts, carbohydrates and any other unidentified factors which may be present in the edible portion of the fruit. The color of the fruit is derived from carotenoid pigments, chlorophyll and possibly flavonoids², whereas the characteristic flavor is obtained from the volatile essential oils found in the peel. All these chemical constituents, plus many others present in smaller quantities, are the result of the combined influences of genetic regulatory mechanisms and the physical, chemical and biological environments to which the fruits are subjected during growth

and after harvest.

Table 1 summarizes some chemical characteristics of several varieties of oranges.

TABLE 1
APPROXIMATE CHEMICAL COMPOSITION OF
ORANGE JUICE (21, 65)

Variety	T.S.S. %	Total Acids as Citric %	pH.	Vitamin C mg./ml.
Parson Brown (Florida)	9.5-11.6	0.55-0.87	3.4-4.3	0.56-0.58
Hamlin (Florida)	8.4-9.6	0.70-0.89	3.4-4.2	0.46-0.50
Homosassa (Florida)	8.1-10.7	0.57-1.5	3.0-4.0	0.47-0.53
Pineapple (Florida)	7.8-12.0	0.75-1.2	3.3-3.9	0.42-0.48
Seedlings (Florida)	8.9-13.1	1.0-2.1	2.8-3.6	0.53-0.54
Valencia (Florida)	10.1-12.5	0.40-1.5	3.1-4.3	0.29-0.45
Valencia (California)	9.1-13.7	0.96-2.02	3.0-3.6	0.41-0.75
Navel (California)	7.2-14.4	0.85-1.3	-	0.29-0.80

As seen from Table 1, the variation in composition of oranges grown in different localities is often so marked that one cannot safely use a general table of composition as a source of precise nutritional information concerning the fruits grown in any one area. In addition, certain nutrients may be destroyed in processing largely because of their sensitivity to the pH of the medium, to oxygen, light and heat or to a combination of these factors. Furthermore, the presence of trace elements and enzymes may enhance these destructive effects. Thus, the methods and materials used in the processing of orange juice are of prime importance for the retention of the maximum nutritive value of the juice.

The economic significance of the marked changes in the utilization of the orange crop reflects consumers' preference and affects growers' returns. In order to utilize any inferior quality fruit profitably, increasing attention should be paid to the processing and the utilization of these oranges.

One obvious way of utilizing this inferior quality fruit is to produce orange juice. It seems imperative, therefore, to investigate the possibilities of simple, economical and practical methods of preserving orange juice of good nutritive value under the conditions prevailing in this area.

In the present study, data have been collected concerning the influences exercised by several methods of

preservation and by the period and conditions of storage on the vitamin C retention, pH, total soluble solids percentage, total acidity and the taste of the juice of three varieties of sweet oranges grown in Lebanon.

RIVIEW OF THE LITERATURE

Little if any data are available on the physiological anatomy of fruits and other factors affecting the quality of expressed juices.⁶⁸ Variety, stage of maturity and growing conditions are known to affect the suitability of fruits for juice production. Furthermore, not all varieties of a given fruit are equally desirable because of the difficulties of preparing juice from them or because the resulting juice is unstable and unpalatable.

The effect of rootstock on the bitterness and other qualities of orange juice was investigated by Marsh.⁵ This worker found that California Navel oranges grown on grapefruit rootstock yielded expressed juice that did not develop a bitter flavor on storage at room temperature, whereas juice from oranges grown on rough lemon rootstock rapidly became bitter after storage. In a similar study, Kefford *et al.*⁶ found that Navel and Valencia oranges, grown on trifoliata rootstock, yielded juice which was free from bitterness and was of outstanding quality as compared with juice from oranges grown on rough lemon rootstock.

According to Biale³ the changes which occur in the

fruit during growth and after harvest affect the quality and stability of the expressed juice. These changes are reflected in both enzymatic and non-enzymatic reactions which occur in the cell sap as a result of the destruction, during extraction of the structural features of the intact cells and the subsequent upset of the dynamic balance among the various constituents present.

It is well known that large differences in the chemical composition of different varieties of fruit as well as within the same variety may occur when the fruits are grown in different geographical areas.²⁴

The effect of processing methods and storage conditions on the quality of fruit juices have been studied extensively and several reviews are available on this topic. 23, 40, 58, 68

According to Guerrant et al.¹⁸ Moschette et al.⁵² and Sheft et al.⁶⁴ there was little change in the nutritive value, including ascorbic acid content, of canned orange juice under normal warehouse conditions if the temperature did not exceed 75° F. Up to 50% loss of ascorbic acid was observed, however, during storage at 80° F over a period of two years.⁴

Brenner et al.⁶ investigated the retention of vitamin C in canned orange juice stored at temperatures of 70° F, 90° F and 100° F for periods up to 18 months. Substantial losses of ascorbic acid occurred after six months

of storage at 90° F and 100° F.

In a study on fresh orange juice, Moore et al.⁵⁰ reported a retention of 97-98% of the original ascorbic acid when the juice was kept at 70° F for 3 days only. After the third day, the juice began to ferment.

Evenden and Marsh¹⁴ studied the effects of various chemicals on the rate of oxidation of ascorbic acid in fresh orange juice. It was found that sulfur dioxide reduced the oxidation rate but that sodium benzoate had little effect.

In a study comparing the effect of glass versus tin packing on the retention of ascorbic acid in orange juice, Moore et al.⁵¹ found that tin-packed juice retained approximately the same percentage of ascorbic acid as glass-packed juice after 6 months of storage at 40° F and 80° F.

Bissett et al.⁴ studied the effect of pasteurization and storage on the quality of sweetened and unsweetened lime juice. According to these workers, juice sweetened up to 45° Brix and unsweetened juice both heated to 200° F developed off-flavors after 4½ months of storage at 80° F.

In a study of the effect of concentration on vitamin retention in orange juice, Hummel and Okey²⁸ found that 70% of the original ascorbic acid was lost after storage for one year at 98° F. Huelin²⁷ reported that concentrated products with a higher than normal

content of fructose and related sugars lost ascorbic acid more rapidly than single strength juice. Curl¹⁰ also studied the effect of concentration on the quality of orange juice and found that ascorbic acid losses increased with increase in concentration under storage temperatures above 80° F. No significant effect of degree of concentration (from 13 to 70 percent soluble solids) was observed at either 40° or 60° F on the development of off-flavors. At 80° F and above, however, the flavor changed considerably, and the deterioration in flavor increased with increase in concentration of the juice under a given set of conditions. Deterioration was related to factors such as development of pressure, darkening, and losses of ascorbic acid and of total sugars. In the same study, it was also found that darkening increased with increase in concentration at storage temperatures of 80° F and above. In addition, significant losses of total sugars were found only at storage temperatures of 80° F and above and increased with increase in concentration with a maximum at 65 percent concentration.

In general, the average retention of ascorbic acid during the canning process is about 97 percent, with an average loss of 1 to 2 percent per month of storage at room temperature.^{14, 16, 35, 44, 46}

Processed orange juice normally undergoes changes in color during storage and develops off-flavors which

may be associated with browning reactions. Pasteurized orange juice in non-refrigerated storage darkens, but this change is masked to some extent by the orange pigments which are suspended in the juice. The darkening occurs in the aqueous phase and is much more evident in filtered juices.^{42, 57}

There is a direct relationship between darkening and ascorbic acid losses in benzoated orange juice as reported by Joslyn.³⁰ Hamburger and Joslyn²⁰ and Joslyn³¹ found that practically all the reduced ascorbic acid disappeared and no other readily oxidizable substances such as sulfur dioxide remained in filtered benzoated juice before darkening occurred. According to Moore et al.⁴⁹ darkening increased with the increase in headspace from 10 to 50 ml in bottled orange juice stored for one week at 120° F. In canned, single strength juice and especially in concentrates, much more ascorbic acid disappears at room temperature than can be accounted for by the oxygen originally present.^{11, 32, 46}

Tressler and Pederson⁶⁹ showed that pasteurized concord grape juice stored in bottles containing substantially no oxygen, underwent very little change even when exposed to light at room temperature for 20 months. When in contact with oxygen, however, the juice became brown within three weeks. There was no difference due to difference in the color of the bottles.

Although little information was found on the effect of light on juice quality, the results of one study [showed that there was no significant light effect on the retention of ascorbic acid or on the general deterioration of bottled orange juice in storage].⁶⁸ Nebesky et al.⁵³ reported that exposure to light caused little deterioration in color but had a bleaching effect on purified pigments of strawberry and current.⁵³ In the same study, pH adjustment between 2 to 4.5 or sugar addition had no effect on color retention in fruit juices during storage.

MATERIALS AND METHODS

The present studies were conducted in the laboratories of the Division of Food Technology and Nutrition, Faculty of Agricultural Sciences, American University of Beirut, during the period of February, 1962 to June, 1963.

EXPERIMENT 1

Selection of varieties

Three commercially important varieties, viz. Bizri, Shammouti and Valencia were selected. Bizri is an early variety whereas Shammouti comes into the market in mid-season and Valencia is a late season variety.

The oranges used in the experiment were purchased from the local market keeping in view to have representatives from all the available lots in the market at the time of purchase.

Preservation methods

Four methods of preservation were used:-

1. Pasteurization
2. Addition of sodium benzoate
- 3.. Addition of sodium metabisulphite
4. Addition of sugar (sugar concentration)

1. Pasteurization

Extracted juice was strained through muslin cloth. Sugar, in the ratio of 3 parts juice to 1 of sugar, was stirred into the freshly strained juice until completely dissolved. The juice mixture was placed in sterilized bottles, capped and pasteurized at 180° F for 30 minutes in the steam retort.

2. Addition of sodium benzoate

Sodium benzoate at the rate of 0.1% was added to the extracted, strained juice. The juice then was immediately sealed in previously sterilized bottles.

3. Addition of sodium metabisulphite

Sodium metabisulphite (0.07%) was added to the strained juice before sealing in previously sterilized bottles.

4. Addition of sugar

The proper amount of sugar (1 to 1 ratio of sugar to juice) was added and the sweetened juice sealed in previously sterilized bottles.

The bottles were sealed with Crown caps in all instances. In the latter three treatments, no pasteurization was performed.

Color of bottle

Plain, amber and green colored bottles were

selected. The size of sample placed in each bottle was 14 ozs.

Storage method

Half of the samples were stored on open shelves and half on closed, dark shelves at room temperature for periods of 3, 6, 9 and 12 months. The storage room was not centrally heated during winter. The maximum room temperature recorded was 92.5° F in summer and the minimum during winter was 47° F.

Observations

The following observations were made on all samples under each treatment initially and after 3, 6, 9 and 12 months of storage:-

1. Ascorbic acid content

For the analysis of ascorbic acid the standard A.O.A.C. method involving visual titration with 2,6-dichlorophenolindophenol was followed.²⁶

2. Total soluble solids (T.S.S.)

Total soluble solids were determined in all samples using an Abbe hand refractometer standardized at 20° C and correction determined on distilled water (A.O.A.C. 1960).²⁶

3. Total acidity

Total acidity of all samples was determined as citric acid by titration with 0.1 N NaOH using phenolphthalein as the indicator (A.O.A.C. 1960).²⁶

4. pH

The pH of the samples was determined electrometrically with a Radiometer pH meter model # 22r.

5. Taste

A taste panel of five members was selected from the staff of the Faculty of Agricultural Sciences. Initially and at the end of each storage period, samples were tasted by these members and graded according to flavor, taste, consistency and color. Results were recorded by each member on prepared grading sheets (Appendix 1). The highest grade was 10 indicating high quality and the lowest was zero. At the time of statistical analysis all the four components, i.e. flavor, taste, consistency and color, which are complementary to each other, were tabulated as one component - taste. The grading was then divided into good, fair and bad; a grade of 5 or above was considered good; 3 to 4, fair, and a zero to 2, poor.

EXPERIMENT 2

A second experiment was conducted during the period of February to June, 1963, to study the effect of headspace on the retention of ascorbic acid in Valencia orange juice. The juice was preserved by addition of sodium metabisulphite and stored in plain bottles on open

shelves at room temperature. Ascorbic acid determinations were performed initially and after 3, 6, 9, 12 and 15 weeks of storage.

In both experiments 1 and 2 the split plot design was followed and three replicates were used in each treatment. A total of 1116 samples were analyzed.

RESULTS AND DISCUSSION

The present studies were undertaken to investigate the effect of length of storage period, method of preservation, method of storage and color of bottle on various chemical and physical characteristics of the juice of three varieties of sweet oranges grown in Lebanon.

The results obtained were statistically analyzed by using the analysis of variance and the 't' test methods.⁵⁶ The critical difference between the treatments was calculated for the 't' test. The results are presented in tabular form at the end of the discussion of each treatment.

The following discussion is based on the statistical treatment of the data. It will be noted that, although significant differences were obtained in many instances, the actual differences were so small as to lose practical importance.

EXPERIMENT 1

Storage period

The lengthening of storage period was found to cause a definite decline in the content of ascorbic acid (Table 2), an increase in total soluble solids percentage (Table 3), an increase in pH (Table 5), and a decrease in the taste quality of the juice (Table 6). Although the mean difference between the percentage of total acids under all the periods of storage was not significant, the data indicate a definite

decrease in the acid value of the juice with increase in the time of storage (Table 4).

It is of value to note that the rate of ascorbic acid loss was faster during the first three months of storage as compared to the rate of loss during subsequent storage periods (Table 2). The same was true of the taste, which deteriorated very fast concurrent with the loss of ascorbic acid during the first 3 months of storage (Table 6).

It is known that canned orange juice loses its nutritive value rapidly under common warehouse conditions when the temperature exceeds 75° F.^{19, 68} Furthermore, during the period in which free oxygen initially sealed in the bottle is being consumed, that is the early period of storage, ascorbic acid deteriorates more rapidly than during later storage.^{8, 12, 15, 62}

The rapid initial decline in ascorbic acid content observed in the present study was undoubtedly influenced by both temperature and the presence of oxygen. The first analyses were made in June after the storage temperature had been relatively high for several weeks. In addition, no attempt was made in the first experiment to eliminate headspace in the bottles; thus the presence of oxygen was introduced as a factor to accelerate the deterioration of ascorbic acid.

The total soluble solids of orange juice include reducing and non-reducing sugars, primarily glucose,

fructose and sucrose. The increase in T.S.S. in this study was probably due to inversion of sucrose to glucose and fructose; a reaction which is known to occur in processed orange juice.²² Also contributing to the higher T.S.S. values would be free sugars released in the hydrolysis of higher carbohydrates such as starch, pentosans and pectin. 59, 60, 61

With the decrease in the total acids the pH value of the juice increased as expected during extended storage at room temperature.⁶⁶

Table 2

Effect of storage on the ascorbic acid*retention in orange juice.

Initial	<u>Storage period in months</u>				Critical difference
	3	6	9	12	
mg./100 ml.					
42.5	24.1	23.4	23.2	22.8	0.096

* Each value represents the average of 60 determinations.

Table 3

Effect of storage on the T.S.S.* contents of orange juice.

Initial	<u>Storage period in months</u>				Critical difference
	3	6	9	12	
%					
10.0	10.5	11.2	11.6	12.2	0.033

* Each value represents the average of 60 determinations.

Table 4

Effect of storage on the total acid* content of orange juice.

Initial	<u>Storage period in months</u>				Critical difference
	3	6	9	12	
%	%	%	%	%	
0.88	0.86	0.79	0.78	0.78	N.S.

* Each value represents the average of 60 determinations.

Table 5

Effect of storage on the pH* of orange juice.

Initial	<u>Storage period in months</u>				Critical difference
	3	6	9	12	
3.9	3.9	4.1	4.1	4.1	0.013

* Each value represents the average of 60 determinations.

Table 6

Effect of storage on the taste* of orange juice.

Initial	<u>Storage period in months</u>				Critical difference
	3	6	9	12	
5.3	3.4	3.4	3.4	3.4	0.372

* Each value represents the average of 60 determinations.

Preservation method

Of the four methods of preservation studied, the addition of sodium metabisulphite resulted in the highest retention of ascorbic acid (Table 7), highest T.S.S. percentage (Table 8), highest total acids (Table 9), highest pH (Table 10), and best taste (Table 11) as compared to juice which was pasteurized, benzoated or preserved by addition of sugar. These observations agree with those of Evenden and Marsh,¹⁴ who, in a study of the oxidation of ascorbic acid in orange juice, found that sulfur dioxide lowered the oxidation rate, whereas sodium benzoate had little effect. Bisset *et al.*⁴ reported also that pasteurized lime juice sweetened to 45° Brix developed off-flavor after 4½ months of storage at 80° F.

Sodium metabisulphite is the acid salt of sulfur dioxide and upon dissolving in water, forms sulfurous acid. This acid serves as an anti-oxidant and inhibits the oxidation of ascorbic acid to L-diketogulonic acid. Although sulfurous acid normally possesses a strong bleaching action it has only a slight bleaching effect on yellow fruit.^{30, 55} Thus a near natural color is retained when this chemical is used.

In the present instance, the sodium metabisulphite appeared to retard the oxidation of ascorbic acid but did not completely stop the reaction. Since the pH of the medium has a definite effect on the oxidation rate of ascorbic

acid,⁴⁵ the losses of the vitamin during extended storage were probably due to the decreased rate of oxidation of sodium metabisulphite within the pH range of 3.6 to 4.6. As reported by Fuller and Crist¹⁷ and Mitchell et al.,⁴⁵ the rate of oxidation of sulfurous acid is independent of pH between 8.8 and 8.2 but decreases between pH 5.9 and 3.2.

The relatively high percentage of T.S.S. in the juice preserved by sodium metabisulphite was probably due to the lower pH which favors the breakdown of higher carbohydrates.^{5, 13, 29}

Table 7

Effect of preservation method on the ascorbic acid* content of orange juice.

<u>Preservation Methods</u>				
Pasteur- ization	Sodiumbenzoate	Metabisulphite	Sugar concent- ration	Critical difference
	mg./100 ml.			
23.3	26.7	31.3	27.4	0.090

* Each value represents the average of 60 determinations.

Table 8
Effect of preservation method on
the T.S.S.* content of orange juice

<u>Preservation Methods</u>				
Pasteur- ization	Sodiumbenzoate	Metabisulphite	Sugar concent- ration	Critical difference
%	%	%	%	
10.9	11.1	11.5	10.9	0.079

* Each value represents the average of 60 determinations.

Table 9
Effect of preservation method on the
total acids* content of orange juice

<u>Preservation Methods</u>				
Pasteur- ization	Sodiumbenzoate	Metabisulphite	Sugar concent- ration	Critical difference
%	%	%	%	
0.60	1.12	1.01	0.54	0.049

* Each value represents the average of 60 determinations.

Table 10
Effect of preservation method on
the pH* of orange juice

Pasteur- ization	<u>Preservation Methods</u>			Sugar concent- ration	Critical difference
	Sodiumbenzoate	Metabisulphite			
4.1	3.6	4.6	4.0	0.231	

* Each value represents the average of 60 determinations.

Table 11
Effect of preservation method on the
taste* of orange juice

Pasteur- ization	<u>Preservation Methods</u>			Sugar concent- ration	Critical difference
	Sodiumbenzoate	Metabisulphite			
3.7	3.6	4.0	3.8	0.206	

* Each value represents the average of 60 determinations.

Variety

Of the three varieties under study, Bizzri, Shammouti and Valencia, the juice of Valencia retained the highest content of ascorbic acid (Table 12), had the

highest T.S.S. percentage (Table 13), highest pH (Table 15), and the best taste (Table 16). The lowest total acids value (Table 14), in the case of Valencia, was probably related to the low initial content of total acids, i.e. 1.02 percent as compared to 1.12 percent of Bizri and 1.20 percent of Shammouti varieties.

Citrus fruits contain a group of distinctive chemical substances which belong to the broad category of flavonoid compounds,² and the pattern of flavonoid distribution within the fruit is associated with taxonomic relationships.⁶⁷ Furthermore, different citrus varieties contain different mixtures of flavonoids. Sweet oranges in general contain hesperidin, eriodictin and rutin, but Valencia oranges are devoid of rutin.^{25, 34} The higher ascorbic acid retention after 12 months in Valencia juice may be related to the absence of rutin, since this is a very apparent difference between Valencia and the other two varieties. It can be speculated that rutin may in some way accelerate the oxidation of ascorbic acid, thus offering an explanation for the lower vitamin C values in the Bizri and Shammouti juice. The exact mechanism of the reaction of flavonoids in general and rutin in particular is not known and requires further investigation.

Another possible explanation of the higher retention of ascorbic acid in Valencia juice may be based on the fact that the pH value was higher than that of the

juice of either Bizzri or Shammouti. This higher pH was perhaps not favorable for the destruction of ascorbic acid, whereas the lower pH of Bizzri and Shammouti possibly enhanced the rate of destruction.^{33, 44}

Table 12
Effect of variety on the ascorbic acid*
content of orange juice

Bizzri	<u>Varieties</u>		Critical difference
	Shammouti	Valencia	
	mg./100 ml.		
28.8	23.3	29.9	0.322

* Each value represents the average of 45 determinations.

Table 13
Effect of variety on the T.S.S.*
content of orange juice

Bizzri	<u>Varieties</u>		Critical difference
	Shammouti	Valencia	
	%		
26.5	26.9	27.9	0.238

* Each value represents the average of 45 determinations.

Table 14
Effect of variety on the total acids*
content of orange juice

<u>Varieties</u>			
Bizzri	Shammouti	Valencia	Critical difference
%	%	%	
0.93	0.96	0.77	0.056

*Each value represents the average of 45 determinations.

Table 15
Effect of variety on the pH* of orange juice.

<u>Varieties</u>			
Bizzri	Shammouti	Valencia	Critical difference
3.55	3.48	4.21	0.159

* Each value represents the average of 45 determinations.

Table 16
Effect of variety on the taste* of orange juice

<u>Varieties</u>			
Bizzri	Shammouti	Valencia	Critical difference
3.1	3.1	5.5	0.227

* Each value represents the average of 45 determinations.

Storage method

The juice stored on closed shelves was found to have a higher content of ascorbic acid (Table 17), higher percentage of T.S.S. (Table 18), and a higher percentage of total acids (Table 19), as compared with the juice stored on open shelves. There was no significant difference between the pH and taste of the juice stored under the two conditions (Table 20 and 21).

Von Loesecke et al.⁷⁰ Loeffler,^{36, 37} and Moore et al.⁴⁷ found that bottled orange juice darkened and developed off-flavors during storage to a greater extent than did juices stored in tin cans. In a study on grapefruit juice, Leuck and Pilcher^{38, 39} found that the retention of ascorbic acid was less in bottled juice than in juice packed in tin containers. They speculated that the greater retention in tin was due to reducing conditions brought about by the presence of the tin. Moore et al.⁴⁸ found little difference, however, in ascorbic acid retention between glass packed and tin packed orange juice after a storage period of six months. These latter findings tend to indicate that the only difference between the glass packed and tin packed orange juice was the effect of light. It seems possible then, that the observations of Leuck and Pilcher were due to the effect of light on the glass-packed juice.

Storage temperature has been found to have a great effect on the retention of ascorbic acid in orange juice. 9, 10, 16, 28, 48, Furthermore, light may also contribute to vitamin C losses. Thus, in the present study, the bottles kept on open shelves were exposed both to light intensities and to the surrounding temperature, whereas the bottles stored on closed shelves were exposed to temperature effect only.

It may be concluded from these observations that light contributed to ascorbic acid destruction, which in turn is associated with the observed increase in T.S.S. percentage and total acids content of the juice.⁴³

Table 17

Effect of storage method on the ascorbic acid* content of orange juice

<u>Method of storage at room temperature</u>		
<u>Open shelves</u>	<u>Closed shelves</u>	<u>Critical difference</u>
mg./100 ml.		
27.1	27.8	0.290

* Each value represents the average of 30 determinations.

Table 18

Effect of storage method on the T.S.S.*
content of orange juice

Method of storage at room temperature

Open shelves	Closed shelves	Critical difference
%	%	
25.6	27.4	0.301

* Each value represents the average of 30
determinations.

Table 19

Effect of storage method on the total acids*
content of orange juice

Method of storage at room temperature

Open shelves	Closed shelves	Critical difference
%	%	
0.83	0.91	0.015

* Each value represents the average of 30
determinations.

Table 20

Effect of storage method on the pH* of orange juice

Method of storage at room temperature

Open shelves	Closed shelves	Critical difference
3.96	3.93	N.S.

* Each value represents the average of 30 determinations.

Table 21

Effect of storage method on the taste* of orange juice

Method of storage at room temperature

Open shelves	Closed shelves	Critical difference
3.4	3.4	N.S.

* Each value represents the average of 30 determinations.

Color of bottles

The juice was packed in plain, amber and green bottles. Data obtained show that the juice packed in plain bottles contained a higher percentage of T.S.S. (Table 23), and a higher percentage of total acids (Table 24); had a higher pH (Table 25) and tasted better (Table 26), as compared to the juice packed in amber and green bottles.

Furthermore, juice packed in plain bottles retained ascorbic acid better than juice kept in green bottles. There was no difference, however, in the ascorbic acid values between the juice packed in plain and amber bottles (Table 22). The differences observed in Tables 22-24, although statistically significant, are too small to be considered of no practical importance.

Tressler and Pederson⁶⁹ did not find any difference in the nutritive value or taste of bottled grape juice due to the difference in the color of the bottles. The present data also lead to the same conclusion.

Table 22
Effect of color of bottle on the
ascorbic acid* content of orange juice

Plain	<u>Color of the bottles</u>		Critical difference
	Amber	Green	
mg./100 ml.			
26.8	26.8	26.0	0.145

* Each value represents the average of 45 determinations.

Table 23

Effect of color of bottle on the T.S.S.* content
of orange juice

<u>Color of the bottles</u>			
Plain	Amber	Green	Critical difference
%	%	%	
25.6	25.4	25.5	0.145

* Each value represents the average of 45 determinations.

Table 24

Effect of color of bottle on the total
acids* content of orange juice

<u>Color of the bottles</u>			
Plain	Amber	Green	Critical difference
%	%	%	
0.83	0.82	0.82	N.S.

* Each value represents the average of 45 determinations.

Table 25

Effect of color of bottle on the pH* of
orange juice

<u>Color of the bottles</u>			
Plain	Amber	Green	Critical difference
0.97	3.94	3.91	0.008

*
Each value represents the average of 45
determinations.

Table 26

Effect of color of bottle on the taste* of orange juice

<u>Color of the bottles</u>			
Plain	Amber	Green	Critical difference
3.2	3.1	3.1	N.S.

*
Each value represents the average of 45
determinations.

Interaction between storage period and preservation method

Data showing the interaction between length of storage period and preservation method are presented in Tables 27 to 31. It was found that juice preserved by addition of sodium metabisulphite retained the highest content of ascorbic acid (Table 27), had the highest total soluble solids percentage (Table 28), highest pH (Table 30) and tasted best (Table 31) during all periods of storage as compared to the juice preserved by the other methods. Although the juice preserved by addition of sodium benzoate had the highest percentage of total acids, the mean difference was not significant when compared to the percentage in the juice preserved by addition of sodium metabisulphite (Table 29).

The rate of ascorbic acid loss in the juice preserved by sodium metabisulphite for all periods of storage was less compared to the loss in the juice preserved by the other methods. The mean difference within the preservation methods for all periods of storage was significant. These results are in agreement with those of Evenden and Marsh¹⁴ who found that orange juice preserved with metabisulphite showed a lower rate of ascorbic acid loss during storage for 18 months as compared to juice preserved with sugar concentration or sodium benzoate.

The mean difference between the total soluble solids percentage of the juice preserved by sodium metabisulphite,

pasteurization, sodium benzoate and sugar concentration was also significant for all the periods of storage (Table 28). As previously discussed, the higher pH, after 6 months of storage of juice preserved with sodium metabisulphite (Table 30) was probably responsible for the higher percentage of T.S.S. (Table 28), and is the result of the lower total acids content of the juice (Table 29).

Table 27

Effect of preservation method on the ascorbic acid* content of orange juice during storage

Preservation methods	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	mg./100 ml.					
Pasteurization	42.5	19.2	18.5	18.3	18.0	
Sodiumbenzoate	42.5	23.8	22.8	22.5	22.0	0.205
Metabisulphite	42.5	29.3	28.5	28.3	28.0	
Sugar concentration	42.5	24.2	23.8	23.6	23.0	

* Each value represents the average of 60 determinations.

Table 28

Effect of preservation method on the T.S.S.*
content of orange juice during storage

Preservation methods	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Pasteurization	10.0	10.5	11.0	11.5	12.0	
Sodiumbenzoate	10.0	10.5	11.2	11.8	12.0	0.178
Metabisulphite	10.0	10.6	11.8	11.9	13.0	
Sugar concentration	10.0	10.5	11.0	11.5	12.0	

* Each value represents the average of 60 determinations.

Table 29

Effect of preservation method on the total acids*
content of orange juice during storage

Preservation methods	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Pasteurization	0.66	0.64	6.60	0.55	0.54	
Sodiumbenzoate	1.25	1.23	1.04	1.04	1.03	0.110
Metabisulphite	0.99	0.98	1.03	1.03	1.02	
Sugar concentration	0.60	0.59	0.51	0.50	0.50	

* Each value represents the average of 60 determinations.

Table 30

Effect of preservation method on the pH* of orange juice during storage

Preservation methods	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Pasteurization	4.18	4.16	4.07	4.06	4.05	
Sodiumbenzoate	3.56	3.58	3.60	3.60	3.61	0.517
Metabisulphite	4.00	4.01	4.96	5.10	5.11	
Sugar concentration	4.15	4.13	3.90	3.90	3.90	

* Each value represents the average of 60 determinations.

Table 31

Effect of preservation method on the taste* of orange juice during storage

Preservation methods	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Pasteurization	5.1	3.0	3.1	3.9	3.9	
Sodiumbenzoate	5.2	3.0	2.9	2.9	2.9	0.464
Metabisulphite	5.9	3.1	2.9	2.9	2.9	
Sugar concentration	5.0	3.9	3.3	2.9	2.9	

* Each value represents the average of 60 determinations.

Interaction between variety and storage period

Valencia orange juice retained the highest content of ascorbic acid as compared to Bizzri and Shammouti during all periods of storage (Table 32). The mean difference between the ascorbic acid content within the same preservation method for all storage periods was statistically significant.

The percentage of total soluble solids in Valencia juice was higher than that in the juice of the other two varieties after the 9th month of storage (Table 33). Higher total soluble solids percentage of Bizzri and Shammouti juice after 3 months of storage was observed, but the mean difference between the T.S.S. percentage of the three varieties after 3 and 6 months was not statistically significant. Valencia juice was also found to have a higher pH (Table 35) and best taste (Table 36) as compared to the other varieties. The higher pH may be due in part to the fact that the Valencia juice had a higher initial pH.

The taste of Valencia juice remained in the 'good' category even after 12 months of storage, whereas Bizzri and Shammouti juice both fell into the 'poor' category after 9 months (Table 36).

The mean difference between the total acids percentage of the Valencia juice and that of Bizzri and Shammouti was not significant for all periods of storage (Table 34). It was observed that the mean difference in total acids percentage due to varieties was so little (Table 14) that

the difference attributed to the interaction between varieties and storage period could not compensate for the significant difference caused by storage. Thus, it can be deduced that with increase in the length of storage period the difference between the total acids percentage due to varieties becomes insignificant.

Table 32
Effect of variety on the ascorbic acid*
content of orange juice during storage

Varieties	Storage period in months					Critical difference	
	Initial	3	6	9	12		
		mg./100 ml.					
Bizzri	47.7	25.0	24.3	24.0	22.0		
Shammouti	41.3	19.2	18.6	18.5	18.0	0.721	
Valencia	38.5	27.9	27.1	27.0	26.9		

* Each value represents the average of 45 determinations.

the difference attributed to the interaction between varieties and storage period could not compensate for the significant difference caused by storage. Thus, it can be deduced that with increase in the length of storage period the difference between the total acids percentage due to varieties becomes insignificant.

Table 32
Effect of variety on the ascorbic acid*
content of orange juice during storage

Varieties	Storage period in months					Critical difference	
	Initial	3	6	9	12		
		mg./100 ml.					
Bizzri	47.7	25.0	24.3	24.0	22.0		
Shammouti	41.3	19.2	18.6	18.5	18.0	0.721	
Valencia	38.5	27.9	27.1	27.0	26.9		

* Each value represents the average of 45 determinations.

Table 33

Effect of variety on the T.S.S.* content of
orange juice during storage

Varieties	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Bizzri	13.5	29.3	29.5	29.7	30.0	
Shammouti	14.5	28.2	29.6	30.2	31.5	0.532
Valencia	12.5	27.7	28.5	34.2	36.5	

* Each value represents the average of 45 determinations.

Table 34

Effect of variety on the total acids*
content of orange juice during storage

Varieties	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Bizzri	1.12	1.02	0.85	0.84	0.80	
Shammouti	1.20	1.11	0.85	0.80	0.75	N.S.
Valencia	1.02	0.79	0.68	0.67	0.60	

* Each value represents the average of 45 determinations.

Table 35
Effect of variety on the pH* of
orange juice during storage

Varieties	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Bizzri	3.80	3.73	3.54	3.53	3.50	
Shammouti	3.60	3.55	3.53	3.52	3.50	0.358
Valencia	4.40	4.33	4.22	4.21	4.20	

* Each value represents the average of 45 determinations

Table 36
Effect of variety on the taste* of
orange juice during storage

Varieties	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Bizzri	4.0	3.4	3.5	2.9	2.5	
Shammouti	4.0	3.4	3.3	2.8	2.4	0.512
Valencia	5.5	5.4	5.2	5.1	5.0	

* Each value represents the average of 45 determinations.

Interaction between storage method and storage period

The data concerning the interaction between storage method and storage period are presented in Tables 37 to 41. The juice stored on closed shelves retained a higher content of ascorbic acid (Table 37), had a higher percentage of total soluble solids (Table 38), and higher total acids content (Table 39), and tasted better (Table 41), as compared to the juice stored on open shelves. The mean difference between the pH of the juice stored on closed and open shelves was, however, not statistically significant (Table 40).

The rate of loss of ascorbic acid was slower in juice stored on closed shelves than in juice kept on open shelves. Also the rate of increase in total soluble solids and total acids percentage was faster in the juice stored on closed shelves than in that stored on open shelves during all periods of storage. Although these differences were statistically significant, no significant difference between the taste of juice stored on closed and open shelves was detected. Thus the mean difference between the tastes was non-significant, but the juice stored on closed shelves had higher values after 12 months of storage.

Table 37

Effect of storage method on the ascorbic acid*
content of orange juice during storage

Method of storage	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	mg./100 ml.					
Open shelves	42.5	23.7	23.0	22.8	22.5	
Closed shelves	42.5	24.4	23.7	23.5	23.0	0.456

* Each value represents the average of 30 determinations.

Table 38

Effect of storage method on the T.S.S.*
content of orange juice during storage

Method of storage	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Open shelves	10.0	28.6	29.0	29.5	30.5	
Closed shelves	10.0	28.9	29.5	33.0	35.0	0.671

* Each value represents the average of 30 determinations.

Table 39

Effect of storage method on the total acids*
content of orange juice during storage

Method of storage	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Open shelves	0.99	0.88	0.77	0.75	0.73	
Closed shelves	0.98	0.96	0.88	0.85	0.82	0.034

* Each value represents the average of 30 determinations.

Table 40

Effect of storage method on the pH* of orange
juice during storage

Method of storage	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Open shelves	4.10	3.98	3.93	3.93	3.92	
Closed shelves	4.10	3.96	3.87	3.87	3.86	N.S.

* Each value represents the average of 30 determinations.

Table 41

Effect of storage method on the taste*
of orange juice during storage

Method of storage	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Open shelves	4.5	3.4	3.4	2.9	2.5	
Closed shelves	4.5	3.5	3.3	2.9	2.6	N.S.

* Each value represents the average of 30 determinations.

Interaction between color of bottles and storage period

Results obtained for the interaction of color of bottles and storage period (Table 42) show that there was a significant mean difference between the ascorbic acid content of the juice kept in plain, amber and green bottles for all periods of storage. The juice in plain bottles retained the most ascorbic acid, and had the highest total soluble solids percentage (Table 43) and the highest pH (Table 45) during all periods of storage. The mean difference between the total acids percentage (Table 44) and taste units (Table 46) of the juice kept in plain, amber and green bottles was not significant. The juice kept in plain bottles, however, tasted better than that stored in amber or green bottles.

Table 42

Effect of color of bottle on the ascorbic acid*
content of orange juice during storage

Color of bottles	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	mg./100 ml.					
Plain	42.5	25.9	25.1	22.3	21.0	
Amber	42.5	23.7	23.0	22.7	21.0	0.327
Green	42.5	22.5	22.0	21.8	20.5	

* Each value represents the average of 45 determinations.

Table 43

Effect of color of bottle on the T.S.S.*
content of orange juice during storage

Color of bottles	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Plain	10.0	28.5	29.2	29.6	30.5	
Amber	10.0	28.3	28.7	29.7	30.1	0.327
Green	10.0	28.5	28.9	29.5	30.5	

* Each value represents the average of 45 determinations.

Table 44

Effect of color of bottle on the total acids*
content of orange juice during storage

Color of bottles	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
	%	%	%	%	%	
Plain	0.89	0.87	0.86	0.80	0.78	
Amber	0.89	0.88	0.85	0.81	0.79	N.S.
Green	0.89	0.86	0.82	0.80	0.76	

* Each value represents the average of 45 determinations.

Table 45

Effect of color of bottle on the pH* of
orange juice during storage

Color of bottles	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Plain	4.00	3.96	3.95	3.95	3.95	
Amber	4.00	3.98	3.89	3.89	3.89	0.002
Green	4.00	3.93	3.86	3.86	3.86	

* Each value represents the average of 45 determinations.

Table 46
Effect of color of bottle on the taste of orange
juice during storage

Color of bottles	<u>Storage period in months</u>					Critical difference
	Initial	3	6	9	12	
Plain	3.7	3.5	3.6	2.9	2.8	
Amber	3.7	3.4	3.5	2.8	2.7	N.S.
Green	3.7	3.6	3.4	2.7	2.6	

*
Each value represents the average of 45
determinations.

EXPERIMENT 2

During the course of experiment 1, it was found that the rate of ascorbic acid loss was much faster during the first three months of storage than during subsequent storage periods. There was no attempt to study headspace effect in experiment 1, even though there was a certain amount of headspace in all bottles. It was decided, therefore, to study the effect of headspace on an ascorbic acid retention during the first three months in bottled Valencia juice preserved with sodium metabisulphite. The rate of loss was evaluated by determining the ascorbic acid content of the juice at the end of every third week of the experimental period.

The results obtained are presented in Tables 47 to 49.

As observed in experiment 1, the length of storage period had a definite effect on the loss of ascorbic acid (Table 47), and the rate of loss was fastest during the first 9 weeks of storage. It was found also that juice bottled without headspace retained a higher percentage of ascorbic acid than juice stored in bottles with $2\frac{1}{2}$ inches of headspace (Table 48). In addition the rate of loss of ascorbic acid in bottles without headspace was slower during all the periods of storage (Table 48). These observations are in agreement with those of Tressler and Pederson,⁶⁹ who demonstrated that the presence of headspace oxygen accelerated the destruction of ascorbic acid during storage. Clayton et al.⁸ reported also that the free oxygen initially sealed in the container causes faster deterioration of vitamin C during the early periods of storage than during subsequent periods. Reister et al.⁶² recommended minimizing the free oxygen content in the container by reducing the headspace in order to obtain better retention of ascorbic acid.

The data in Table 49 show that, for all periods of storage, the ascorbic acid content was significantly higher in samples bottled without headspace as compared to that of samples in bottles with headspace (Table 49). The mean difference between the ascorbic acid content within the same headspace under all storage periods was significant. Juice

bottled without headspace lost ascorbic acid less rapidly than juice in bottles with headspace.

Table 47

Effect of storage on the ascorbic acid*
content of Valencia juice

Initial	Storage period in weeks					Critical difference
	3	6	9	12	15	
mg./100 ml. juice						
39.5	37.3	35.5	31.0	30.2	29.2	0.475

* Each value represents the average of 36 determinations.

Table 48

Effect of headspace on the ascorbic acid*
content of Valencia juice

With headspace	Without headspace	Critical difference
mg./100 ml. juice		
31.91	35.69	0.219

* Each value represents the average of 36 determinations.

SUMMARY AND CONCLUSIONS

Two experiments were conducted to study the influence of different methods of preservation and periods and conditions of storage on the chemical composition of the juice from three varieties of sweet oranges grown in Lebanon.

In Experiment 1, juice from Bizzri, Shammouti and Valencia oranges was preserved by pasteurization or by addition of either sugar, sodium benzoate or sodium metabisulphite. The treated samples were stored in plain, amber or green bottles, on open or closed shelves at room temperature. Initially and after 3, 6, 9 and 12 months of storage, the following observations were made on each sample:

1. (1) ascorbic acid content, (2) percentage total soluble solids (T.S.S.), (3) percentage of total acids, (4) pH and (5) taste.

The second experiment was designed to study the retention of ascorbic acid in Valencia orange juice preserved with sodium metabisulphite and stored in plain bottles, on open shelves at room temperature for 15 weeks. Ascorbic acid was determined initially and after every third week of storage.

According to the statistical analysis the results of Experiment 1 can be summarized as follows:

1. Juice preserved with sodium metabisulphite retained the highest content of ascorbic acid, had the highest T.S.S., total acids and pH, and tasted better throughout the entire storage period than juice preserved by any one of the other 3 methods.
2. Of the three varieties of sweet oranges studied, Valencia orange juice retained the highest content of ascorbic acid, and had the highest T.S.S. percentage, pH value, and the best taste. The total acids percentage was lowest after 12 months of storage in Valencia juice.
3. The juice stored on closed shelves retained a higher content of ascorbic acid and had a higher percentage of T.S.S. and of total acids as compared to the juice stored on open shelves. The difference between the pH and taste of the juice stored on closed and open shelves was not significant.
4. There was no significant difference between the ascorbic acid content of juice kept in plain bottles and in amber colored bottles, but the juice kept in green bottles lost more ascorbic acid than juice stored in either plain or amber bottles. A higher T.S.S. percentage, higher percentage of

total acids, higher pH and better taste were observed in juice kept in plain bottles as compared to that stored in amber and green bottles.

It should be pointed out that in many instances the critical differences were so small that they could not be considered of much practical value.

The results of the second experiment showed that the overall loss of ascorbic acid was greater in juice stored with headspace than in juice kept in bottles without headspace. Furthermore, the rate of loss with headspace was more rapid than in bottles in which headspace was eliminated.

The data in general indicated that, under the conditions of this experiment, a processed product of good flavor, nutritive value and storage potential could be obtained by preserving Valencia orange juice with sodium metabisulphite.

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Appendix 1. Taste Panel Grading Sheet

Food Technology

SAMPLE NO.

Taste Panel

Date:

- | | |
|---------------------------|---------------|
| 1. TASTE | <u>Grades</u> |
| a. Good | a. |
| b. Fair | b. |
| c. Bad | c. |
| 2. ODOR | <u>Grades</u> |
| a. Good | a. |
| b. Fair | b. |
| c. Off | c. |
| 3. CONSISTENCY | <u>Grades</u> |
| a. Thick | a. |
| b. Medium | b. |
| c. Thin | c. |
| 4. COLOR | <u>Grades</u> |
| a. Normal (Light yellow) | a. |
| b. Bright (Bright yellow) | b. |
| c. Brown | c. |

Name of Panel Member

1. Judge each quality separately.
2. Give grades from 1 to 10.