MUHAMMAD KHALIL, MUHAMMAD RAMZAN, AMJAD ALI and RIAZ AHMAD RIAZ

Department of Food Technology, University of Agriculture, Faisalabad

(Received January 23, 1979; revised May 5, 1979)

Abstract. The introduction of comminuted beverages is of recent origin. These types of beverages are found to be not only more nutritious but also superior to the conventional squashes in colour, aroma and cloudiness. The comminuted beverages are better protected against oxidative spoilage during extended period of storage. This is one of the first attempt on laboratory scale in respect to preparation techniques and to see the storage stability with a view to find the possibilities of introduction of comminuted lime squash in Pakistan.

Citrus fruits occupy a prominent position in the fruit Industry of Pakistan. These fruits are considered to be a rich source of ascorbic acid, pectin, carotene, citric acid, peel oil and some of the minerals like calcium, iron, phosphorus etc. Incorporation of citrus peel in the comminuted drink causes an appreciable increase in ascorbic acid content as citrus peel contains more than twice the amount of ascorbic acid than does the juice.

The limes (*Citrus aurantifolia*) mature in the months of July/August when no other citrus fruits are available in the market. Usually in our country lime fruit is utilized in the manufacture of squash, cordial, and barley water.

The introduction of comminuted beverages is of recent development. The comminuted beverages are said to be better protected against oxidative spoilage during extended period of storages.^{1,13,17} These type of beverages are considered more nutritious not only to the squash prepared by conventional methods but also to synthetic beverages, i.e. Coca Cola, Fanta, Seven Up etc.

The suspended solids are more or less stablised with little clearing of juice in the absence of enzymatic action. The material which remained in more or less permanent suspension was referred to as "cloud" of juice. The citrus juice becomes "watery" when this colloid is degraded which ultimately resulted in upsetting of the colloidal balance and settling of the suspended material as well as rapid separation of water insoluble solids from the pulp.14,16

It is reported that stability of turbid fruit juices can be achieved by short time heat treatment at 87° so as to inactivate pectolytic enzymes or by effective homogenization or by increasing the viscosity through the addition of hydrocolloids.^{5,6}

The ascorbic 'acid 'content of flavedo is much higher than that of the albedo portion of the citrus peel.⁷ It is observed that the ascorbic acid content decreases slightly during processing and there is no pronounced change during first 15 days of storage, but thereafter a rapid decrease is recorded.⁹ A slight loss in acidity and a gradual increase in pH is observed in orange squashes during storage.¹¹ The reducing sugars increases during storage in orange juice, but the pectin content remaine's constant throughout the storage period.²

In canned cling peaches an increase in ^O Brix reading during storage is observed. A similar increase in soluble solids in canned oranges is recorded during storage at ambient temperature.⁸,12

The first detailed account of comminuted citrus beverages was reported in 1963. These novel type drinks were made from the whole fruit by reducing its entire contents including the peel, pith, and seed, to a state of particle subdivision to ensure a homogeneous product having a stable emulsion.⁵

A novel process consisted in steaming the whole oranges until quite soft in a conical pressure cooker with a valve at bottom and blowing out the contents and; subsequently milling and finishing. The final comminuted product so obtained was found to be superior in quality as compared with the product prepared by the conventional method.³

Upto now no such products are being produced in Pakistan. The present project was, therefore, designed to develop from the whole fruit a product which might be superior to conventional squashes in colour, aroma, nutritive value and storage stability. These studies were also conducted on the possibilities of incorporation of more pectin and essential oil through the portions of peel in the product which give a heavier body as well as a dense and stable cloud alongwith a rich enduring flavour.

Experimental

Technique of Preparation of Comminuted Lime Juice and Squash. The sound and properly mature limes were purchased from the market. After washing in running water overripe or damaged fruits were removed. The juice was extracted as under.

1. By Conventional Method. The juice was extracted with the help of wooden juice squeezer after halving the individual fruit and strained through a muslin cloth.

2. By the Process of Comminution. The juice was extracted in the following two ways after comminuting the whole fruits.

(a) From the Un-blanched Whole Fruit. The whole fresh fruits were reduced to a homogeneous mass by using an Osterizer. The juice was obtained with the help of a basket press.

(b) From Blanched Whole Fruit. The whole fruits were heated in water so as to obtain a temperature of 87° at the centre of the fruit. After cooling rapidly (winthin 5 minutes) to room temperature the fruits were reduced to a uniform mass in an Osterizer. The juice was separated from the coarse pulp by straining through a muslin cloth kept in the basket press.

The lime squash was prepared from the juices so obtained in each lot separately by adopting the recipe given below.

Juice 19.70%, sugar 40.00%, citric acid 00.30%, water 40.00%, SO₂ 350 ppm.

The squash thus prepared from each treatment was filled in seven ounce capacity bottles, sealed with crown cork and stored at ambient temperature.

Squash Evaluation. The squash samples kept at ambient temperature were analysed after 0, 15, 90, 150, and 240 days for acidity, pH, ascorbic acid, Brix, pectin, and reducing sugars by adopting methods of Ruck.¹⁵

By determining per cent transmittance of squash samples without any pectinol treatment with the help of spectrophotometer, the density of colloidal suspension was studied. The degree of settling after each storage interval was determined in centimeters with the help of a divider by measuring the distance between bottom and the point of settling in each bottle. The average of three readings is recorded and used as an indication of the extent of settling.

The comminuted squash was also evaluated organoleptically for colour, taste and flavour during storage intervals of 0, 90, 240 days by numerical scoring method.¹⁰ The judges were provided a score sheet to record the scores.

Results and Discussion

This study was conducted to find the feasibility of production of comminuted lime squash in Pakistan. The squash, prepared from juice extracted by different methods in laboratory scale, was stored at ambient temperature (ranged from 45° in summer and 10° in winter) for 240 days. The results obtained are discussed with respect to ascorbic acid, acidity and pH, total soluble

solids, pectin, reducing sugars, cloud stability, and degree of settling as well as organoleptic characterisitcs.

Ascorbic Acid. The results in Table 1 indicate that during a storage period of 240 days the ascorbic acid content in lime squash ranged from 3.84 to 0.21 mg/ 100 ml prepared from the juice extracted by conventional method, from 13.05 to 0.31 mg/100 ml prepared from the juice extracted by the process of comminution without pasteurization and 6.72 to 0.30 mg/100 ml when prepared from the juice extracted hy comminution after the fruit was blanched in hot water. There was a substantial loss of ascorbic acid in the all three treatments upto 45 days as compared to the remaining period of storage.

The statistical analysis of the data given in Table 5 for ascorbic acid content showed a non-significant effect for the methods of juice extraction whereas storage intervals are highly significant.

In freshly prepared squash the ascorbic acid content was the lowest in case of juice extracted with conventional method and the highest in case of juice extracted after comminution, but without pasteurization. This increase in ascorbic acid content may be due to additional ascorbic acid incorporation in juice from the peel of the fruit. A decrease in ascorbic acid was recorded in squash prepared from juice extracted by comminution process after the fruit has been blanched in hot water. This loss of ascorbic acid was probably due to heat treatment of juice in the squash.

These results are in agreement with that of others.⁷ A decrease in ascorbic acid content in all the treatments during storage was observed. This rapid decrease in ascorbic acid might be due to high temperature of storage during summer. The trend in loss of ascorbic acid was supported by other studies.⁹,12

Acidity and pH. The Table 2 indicates the effect of storage on the acidity of squash prepared by different treatments. The initial acidity was observed to be 1.65% in squash prepared with conventional method; 1.66% in comminuted squash without heat treatment; and 1.66% in case of comminuted sample of squash made from blanched whole lime fruit in hot water. After 240 days of storage the acidity ranged from 1.58 to 1.59% in all the three differently treated samples of squash. The storage intervals differed highly significantly for acidity as shown in the Table 5.

The pH values in conventionally prepared sample, comminuted and hot water blanched comminuted samples of squash ranged from 2.66 to 2.71, 2.65 to 2.70 and 2.65 to 2.70 respectively during a period of 240 days of storage. The statistical analysis of data in Table 5 for pH showed highly a significant effect for storage period.

A gradual decrease in acidity with a corresponding slow increase in pH values were observed during a storage period of 240 days in all the samples of squash prepared

268'

PREPARATION AND STORAGE STABILITY OF COMMINULATED LIME SQUASH

	•	Stor	age in days			
Treatments	0	15	45	90	150	240
1*	3.84	3.77	2.10	0.53	0.23	0.21
2**	13.05	6.56	3.77	0.52	0.34	0.31
3***	6.72	4.99	2.88	0.54	0.30	0.30

TABLE 1. SHOWING THE EFFECT OF STORAGE ON ASCORBIC ACID CONTENT mg/100 ml IN. COMMIMUTED LIME SQUASH PREPARED UNDER DIFFERENT TREATMENTS.

* = Squash prepared by conventional method; **= Comminuted squash prepared from the whole fresh fruit; *** =Comminuted squash prepared after blanching the whole fruit.

> TABLE 2. SHOWING THE EFFECT OF STORAGE ON PERCENTAGE ACIDITY AND pH IN COMMINUTED LIME SQUASH PREPARED UNDER DIFFERENT TREATMENTS.

Treatments	0		15		Storage 45	1 C			150		240	nanonen an
	а	b	a	b	а	b	a .	b	a	b	a	b
1*	1.65	2.28	1.64	2.29	1.63	2.30	1.60	2.32	1.60	2.35	1.59	2.40
2**	1.66	2.65	1.65	2.65	1.64	2.67	1.63	2.68	1.60	2.70	1.58	2.70
3***	1.66	2.75	1.64	2.76	1.63	2.79	1.62	2.80	1.60	2.80	1.58	2.85

*= = Squash prepared by conventional methods; ** = Comminuted squash prepared from the whole fresh fruit;

** = Comminuted squash prepared after blanching the whole fruit., a = acidity and b = pH.

differently. The decrease in acidity was found to be in agreement with the results reported for orange squash.¹¹ This reduction in acidity during storage may be due the chemical reactions of various organic acids with other contituents of squash.

Degree of Brix. The initial Brix ranged from 41.5 to 42.0 in all the three treatments. A slight increase in^o Brix was observed under all the treatments during storage period of 240 days. These results agreed with the results of others. 8,12

The statistical analysis of data in Table 5 indicated that the effect of different treatments and storage intervals were nonsignificant. Pectin. Table 3 indicates the results on the pectin content under the different treatments of preparation of lime squash during storage period of 240 days at ambient temperature. A regular and slight decrease in pectin content was recorded in case of conventionally prepared lime squash and also in comminuted sample prepared without heat treatment during a storage period of 240 days as the range of pectin content was 0.11-0.05% and 0.78-0.48% respectively. But in case of comminuted lime squash prepared by blanching the whole fruit the pectin content increased from 0.78 to 0.83% during storage.

The higher pectin content in case of comminuted squash was due to incorporation of peel in the juice.

TABLE 3. SHOWING THE EFFECT OF	F STORAGE ON PECTIN CONTENT % IN COMMINUTED LIME SQUASH
PREPAR	RED UNDER DIFFERENT TREATMENTS.

	Storage in days								
Treatments	0	15	45	90	150	240			
1*	0.11	0.09	0.09	0.08	0.06	0.05			
2**	0.78	0.69	0.66	0.66	0.58	0.48			
3***	0.78	0.78	0.79	0.81	0.81	0.83			

* = Squash prepared by conventional method; ** = Comminuted squash prepared from the whole fresh fruit; ***=Comminuted squash prepared after blanching the whole fruit. Heating caused a more increase in soluble pectin content in case of comminuted lime squash prepared after blanching of fruit and also protected the pectin from enzymatic degradation. This increase in pectin content may be explained in parts by the formation of water soluble pectin from the insoluble pectin fraction during storage. The presence of pectic enzymes in case of first two treatments caused a substantial loss in pectin content. These results are comparable with observations of other.²

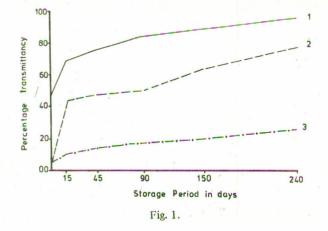
The statistical analysis of data as reported in Table 5 for pectin content was highly significant for the different treatments of juice extraction and non-significant for storage intervals.

Cloud Stability. The cloud stability was determined by measuring per cent transmittance of light with help of Spectronic 20. The higher the percentage of light transmitted, the higher would be the cloud loss and vice versa. To evaluate the squash for cloud stability under different treatments of preparation, per cent transmittance was recorded from the sample drawn at a central point in bottles when kept undisturbed.

The Fig. 1 indicates the trend of per cent transmitrancy recorded from samples of the bottled squash. Initially in case of conventional preparation treatment, the per cent transmittance value was 50 and in other two squash samples the values were 6 and 6 for both the treatments of comminution. The final reading recorded ranged from 26 to 98% after 240 days of storage for all the three squash samples prepared differently.

Table 5 indicates the statistical analysis of the results given in above Figure. These results are highly significant for the methods of juice extraction as well as storage intervals.

It is quite obvious from the above results that initially in squash prepared by conventional method, there is less colloidal matter present as indicated by high percentage of transmittance reading than those of other both the comminuted squash samples. The lower transmittancy values in case of comminuted squash samples prepared either with or without blanching treatment indicate the

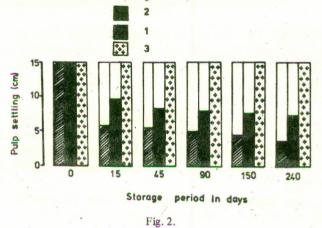


presence of high amounts of colloidal matter. This increase in cloudiness is due to incorporation of more colloids by the process of comminution.

During storage a clear and more separation of cloud into two parts, i.e. less denser towards the top and more denser towards the bottom of comminuted preparations. This instability of cloud can be attributed to the presence of enzymes which caused degradation of pectic substances. In case of comminuted squash prepared from hot water blanched whole fruit the cloud remained stable and dense during storage. This stability of cloud is due to blanching treatment which inactivated the enzyme system. The trend for the results on cloud stability is in agreement with the results of others. 4,6,13,14

Degree of Settling. The data on the degree of settling of squash prepared by different treatments during various storage periods have been illustrated in Fig. 2. The degree of settling was determined in centimetre by measuring the distance between the bottom and the point of separation in each bottle (constant height fill = 15 cm) kept undisturbed.

The rate of pulp settling increased with the increase of storage period in case of squash prepared from unheated fruit in both the conventional and comminuted processes as in these cases the maximum settling was 11.25 cm and 7.20 cm respectively.



In case of conventionally prepared squash the rate of settling was maximum as compared to unheated comminuted squash during storage period of 240 days. There was observed to be no obvious settling in case of comminuted squash sample prepared from hot water blanched fruit even upto 240 days of storage period. Although the density of cloud was found to be lesser towards the top and greater towards the bottom of filled bottles. The highest settling rate in case of conventionally prepared squash sample can be attributed to the presence of active enzymes and to large particle size of suspended pulp. The reduction of particle size of suspended pulp to homogenous mass due to the process of comminution decreased the rate of settling of pulp as compared to conventional squash during storage.

The maximum cloud stability in case of comminuted squash prepared from hot water blanched fruit is found to be by the complete inactivation of enzyme system due to blanching treatment and to a fine state of particle subdivision -of -suspended colloids caused by the process of comminution.

The partciles of the size of less than 2 mu constituted a stable cloud. The fraction consisted of needle like crystals of hesperidin, chromoplastids, amorphous particles and oil globules on the rag particles enhanced their stability in suspension by decreasing their density.⁶

The results reported in Table 5 indicate that the methods of juice extraction treatments as well as storage intervals differ significantly for the degree of settling.

These results on pulp settling are comparable with the observations of other workers.4,5,14

Reducing Sugars. The results in the Fig. 3 reveal the effect of storage under different treatments on reducing sugars. Initially the range of reducing sugars varied from 3.95 to 7.43% while after a storage period of 150 days it varied from 42.64 to 43.02% under all the different treatments. During storage reducing sugars increased even 9 to 19 times as compared to initial contents. The conversion may be attributed to high temperature during storage (45°) in summer with associated effect of high acid content of samples as confirmed by the other findings.²

The statistical analysis of data given in Table 5 showed a significant difference for methods of juice extraction and highly significant effect for storage intervals.

Organoleptic Evaluation of Squash. The samples of lime squash prepared by different treatments were evaluated organoleptically by score ranking test method for colour, taste and flavour attributes during storage intervals of 0, 90 and 240 days. The squash samples, diluted to desired taste with iced water to constant volume, were evaluated by a permanent panel of eight judges. For colour the mean score values were in the range of 5.8 to 6.1 out of the total score of ten for standard of excellence for all the three treatments.

The taste mean score values were from 6.2 to 6.4; 8.4 to 9.0 and 8.4 to 9.5 for conventionally, unblanched comminuted and blanched comminuted samples of squash respectively during storage of 240 days.

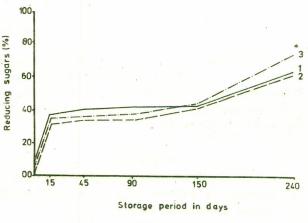


Fig. 3.

The mean score values for flavour varied from 5.6 to 5.8 for the conventional sample of squash whereas in case of comminuted squash it varied from 8.9 to 9.1 in unblanched; and 8.7 to 8.9 in blanched whole fruit, as explained in Table 4.

The higher score values in case of both the comminuted samples were recorded for the taste and flavour attributes as compared to those of (control) conventionally prepared squash sample. This better flavour and taste in case of comminuted squash samples were due to incorporation of more flavedo portion containing lime oil by the process of comminution.

A tocopherol like substance rich in antioxidant activity was isolated from the flavedo of the citrus fruits. This substance might be the cause of remarkable stability of flavour in the comminuted drinks.¹⁵

TABLE 4. SHOWING THE MEAN NUMERICAL SCORE VALUES OF EIGHT JUDGES FOR ORGANOLEPTIC CHARACTERISTICS OF COMMINUTED LIME SQUASH PREPARED UNDER DIFFERENT TREATMENTS DURING STORAGE AT AMBIENT TEMPERATURES.

Storage period		Colour Treatment	S		Taste Treatments			Flavour Treatments		
in days	1	2	3	1	2	3	1	2	3	
0	6.0	6.1	5.9	6.4	9.0	8.5	5.8	9.1	8.9	
90	6.0	6.0	6.0	6.3	8.6	8.4	5.7	9.0	8.8	
240	6.0	5.8	6.0	6.2	8.4	8.4	5.6	8.9	8.7	

1 = Squash prepared by conventional method; 2 = Comminuted squash prepared from the whole fresh fruit;

3 = Comminuted squash prepared after blanching the whole fruit.

M. KHALIL, M. RAMZAN, A. ALI and R.A. RIAZ

TABLE 5. RATIO VALUES FOR THE VARIOUS CONSTITUENTS IN COMMINUTED LIME SQUASH AS AFFECTED BY STORAGE INTERVALS AND TREATMENTS.

Due to	Ascorbic acid	Acidity	pH	Brix	Pectin	Reducing' sugars	Transmi- ttancy	Degree of settling	Colour	Taste	Fla- vour
Treatments	2.56NS	229.91**	178.00NS	2.72NS	229.83**	3.99*	68.46**	367.09**	2.97NS	1.343**	3.11**
Storage	8.85**	5.51**	16.50**	1.09NS	1.02NS	152.00**	8.93**	12.21**	0.50NS	1.316*	1.49*

N.S. = Non-significant: * = Significant: ** = Highly significant.

Acknowledgements. The authors wish to express their thanks to Dr. Muhammad Shafiq Chaudhry, Chairman, Department of Food Technology, University of Agriculture, Faisalabad, for providing all possible help and facilities for conducting this research.

References

- 1. Anonymous, Food Manufactures, 34, 99 (1959).
- M. Ali and R. Rahman, University of Agriculture Res. Studies, (Fasailabad), 1, 118 (1965).
- 3. J.B.S. Braverman and A. Levi, Food Technol., 14, 106 (1960).
- 4. R.H. Briggs and J.E. Pollard, Proc. Fla. State Hort. Soc., 83, 314 (1970).
- 5. V.L.S. Charley, Food Technol., 17, 33 (1963).
- 6. F. Engelhardt, Food Manuf., 39, 72 (1964).
- H. Habib and Z. Ismail, Agric. Res. Rev. (Cairo) 37, 514, (bibl. 9) (1959), cf. Hort. Abstr. 6918 (1961).
- 8. B.W.T. Kanujoso and B.S. Luh, Food Technol., 21,

139A (1967).

- A. Karim and R. Rahman, Pakistan J. Agri. Sci., 4, 215 (1967).
- 10. J.K. Krum, Food Engg., 27, 83 (1955).
- 11. A.A. Malik, M.Sc. Thesis, University of Agriculture, Faisalabad (1965).
- M. Maleki and S. Sarkissian, J. Sci. Food and Agri., 18, 501 (1967).
- 13. P.V.S. Rao, P.S.R.K. Prasad, G.N. Rao and N. Giridhar, Indian Food Packer, 22, 1 (1968).
- 14. A.H. Rouse and C.D. Atkins, Food Technol., 6, 291 (1952).
- 15. J.A. Ruck, Chemical Methods for Anaylysis of Fruit and Vegetable Products (Canada Department of Agriculture, Pub. No. 115, 1963).
- 16. J.W. Stevens, D.E. Prithett and W.E. Baier, Food Technol., 4, 469 (1950).
- 17. S.V. Ting and W.F. Newhall, J. Food Sci., 30, 57 (1965).