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FLAVOR FORTIFICATION AND STORAGE OF FOAM-MAT DRIED GRAPEFRUIT CRYSTALS[1]

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ABSTRACT

The effectiveness of a number of different flavoring additives with foam-mat dried grapefruit crystals has been studied. These included sweetening agents as well as grapefruit flavoring agents. It was found that calcium cyclamate at a level of 0.3% or a combination of calcium cyclamate with sodium saccharin at a level of 0.2% was equivalent in sweetness to a grapefruit sample where the Brix/acid ratio had been adjusted to 13 by the addition of sucrose. When these samples were compared by storage the sucrose sweetened sample was found to have a storage stability of nine weeks before a detectable difference occurred at 85° F, whereas the synthetic sweetener samples had developed detectable differences at six weeks. Among other flavoring agents studied were grapefruit cold-pressed peel oil, essence oil, a commercial grapefruit flavoring, and "locked-in" oil. Although satisfactory initial flavors were obtained with all of these materials, none of the first three had a storage stability equivalent to that of the "locked-in" oil.

In an additional study to determine the level of grapefruit oil which would be preferred by inexperienced tasters it was found that considerably higher oil levels were preferred than had been expected. The preferred oil level found in this study was 0.012%. It was also shown that the higher oil levels appear to be more appealing to non-users, particularly. A study with freeze-dried grapefruit juice used as a flavoring additive indicated that when added at as low as 10% level, freeze-dried grapefruit juice is an effective flavor enhancer. Based on results of this study, it would appear that "locked-in" oil crystals are probably the most suitable flavoring agent for grapefruit foam-mat dried crystals, especially from the standpoint of stability.

During the past few years considerable interest has developed in the use of the foam-mat drying process for the production of citrus crystals. Although considerable problems were encountered with storage stability in orange crystals, the production of grapefruit crystals has been developed to a point where they appear to be commercially feasible (2). Relatively large quantities of foam-mat dried grapefruit crystals have been produced during the past two years on pilot-plant foam-mat drying equipment at the Fruit and Vegetable Products Laboratory in Winter Haven. A large number of grapefruit crystal samples were distributed throughout the citrus industry during the past year in a trade journal (1). Considerable commercial interest has been shown through requests for samples and technical information. Information was needed as to types of flavoring agents which might be used in conjunction with grapefruit crystals for flavor fortification.

Recently, considerable concern has also been shown in the use of grapefruit products as diet foods. Because of this, many inquiries have been received pertaining to the compatability of various diet sweetening agents with grapefruit crystals and concerning the use of sugar-sweetened grapefruit crystals. Before commercial use of these mixtures could become practial, storage data were needed concerning the relative storage stability of sweetened grapefruit crystals as compared to unsweetened

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References to specific commercial products do not constitute endorsement.

grapefruit crystals. Information was also needed as to the relative storage stability of grapefruit crystals sweetened with artificial diet sweeteners as compared to grapefruit crystals sweetened with sugar.

A study was made in our Laboratory to resolve some of these questions. A number of grapefruit flavoring agents were tested in conjunction with grapefruit crystals and storage tests on these were run at 85° F. Previous storage studies on grapefruit crystals prepared with "locked-in" peel oil have shown that the average storage life before a detectable difference occurs in grapefruit crystals is approximately 6 months at 70° or 12 weeks at 85° F (3). Because of the extended times involved in 70° F storage, the tests presently reported were all carried out at 85° F storage. Based on past experience, it would be probable that storage times at 70° F would be approximately 2 to 3 times the storage life at 85° F.

Experimental

All of the grapefruit crystals used in this study were prepared on a crater-type foam-mat drier by the method described by Berry, et al. (2). They contained 0.45% methylcellulose as a single additive. They were prepared from 50° Brix grapefruit concentrate having a Brix/acid ratio of 10.5. All samples had a high initial quality, and had initial moisture content of 1% or less. All samples were sealed in air, at atmospheric pressure.

Grapefruit Flavoring Agents

Peel Oil: Commercial cold-pressed grapefruit peel oil was added to the grapefruit concentrate at several different levels. It was found through determination of oil content in the grapefruit crystals, that 60% of the peel oil added to the concentrate was recovered in the final crystals as determined by the Bromate Titration method (5). Grapefruit samples were made with several different oil levels and these were compared by triangle taste tests with a control sample containing "locked-in" oil.

Essence Oil: A sample of grapefruit oil decanted off the aqueous essence recovered from an essence stripping system at the Citrus Experiment Station, Lake Alfred, Florida, was added to the grapefruit concentrate in the same manner as the grapefruit peel oil described above (6).

Commercial Grapefruit Flavoring: A sample of grapefruit flavoring which was supplied by Polak's Frutal Works, Inc. was added to foam-mat dried grapefruit crystals at several different levels and compared to samples flavored by other materials.

"Locked-in" Oil: As a control sample, grapefruit crystals containing 0.005% grapefruit peel oil as "locked-in" oil supplied by Orange Products Div., Sunkist Growers, Ontario, California, were used as described previously (3), for comparison to samples flavored by other materials. This is grapefruit peel oil encapsulated in crystalline sugar by a patented commercial process.

Sweetening Agents

Grapefruit crystals were used with three different sweetener agents as follows:

Sucrose Control: The Brix/acid ratio of the grapefruit crystals was adjusted to 13 ± .5 by the addition of 35.1 gm of pure sucrose for each 130 gm of grapefruit crystals.

Cyclamate: Calcium cyclamate (Abbott Laboratories, North Chicago, Illinois) was added to grapefruit crystals at various levels and tested in comparison with samples sweetened with sucrose until a level was found at which there was no detectable difference.

Cyclamate-saccharin: A mixture of calcium cyclamate and sodium saccharin (1/10) (Abbott Laboratories, North Chicago, Illinois) was added to grapefruit crystals and the samples tested until a level was found where there was no detectable difference between this sample and the sucrose sample listed above. When equivalent levels of sweetness were found for these three agents, samples were prepared and storage tests were carried out at 85° F for detectable differences.

Oil Levels

Commercial "locked-in" peel oil was added to foam-mat dried grapefruit crystals at the levels of 0.0075, 0.010, and 0.012%. These samples were sent to the Statistical Reporting Service, Washington, D. C., where they were evaluated by a group of untrained tasters. These tests were carried out among both users and nonusers of grapefruit juice. The samples were evaluated by preference score on a Hedonic Scale rating from 1 to 10. A score of 1 represented a very poor quality sample which was disliked and a score of 10 represented an extremely liked high-quality sample. Overall mean scores were

determined as well as separate mean scores among users and among non-users.

TASTE TESTS

With the exception of the oil level samples mentioned above, all samples were tested by the triangle or paired comparison methods described by Boggs and Hanson (4). Samples of reconstituted juice from foam-mat dried grapefruit crystals to which the respective flavoring agent under study had been added were compared for detectable flavor differences. From the respective powders, grapefruit juice was reconstituted to 10.5° Brix by adding 42° F water. These samples were adjusted so that there was no more than 1° F difference of temperature and presented to a panel of 24 experinced tasters. For tests to determine equivalent level of grapefruit flavoring or equivalent sweetness, the level of the respective flavoring agent was adjusted until there was no detectable difference between the experimental and the control sample in a triangle test. For storage tests, samples were placed in storage at 85° F and tested weekly until there was a significant detectable difference between the sample stored at 85° F and a similar sample stored at 0° F. For all triangle and paired comparisons, differences were considered significant when results reached a confidence level of 95% or better.

RESULTS AND CONCLUSIONS

The results of the study of equivalent sweetness of three different sweetening agents are shown in Table 1. With the grapefruit crystals used, sucrose was required at a level of 27%, (w/w) to adjust the Brix/acid ratio to 13. This sample served as a control. Calcium cyclamate was tested at levels from 0.1% to 0.7%. It was found that the level of 0.3% imparted sweetness to the grapefruit crystals which was not detectably different from the control. A calcium cyclamate and sodium saccharin combination (1/10) was also tested at several different levels and it was found that the equivalent sweetness for a 13 ratio was in this case 0.2% of the combination. Batches of the three grapefruit crystal samples of equivalent sweetness were placed in storage at 85° F and tested weekly against respective control samples stored at 0° F in triangle taste tests. The results of these storage tests are indicated in Table 2. At the end of six weeks storage time the samples sweetened with calcium cylamate had developed a detectable difference for their respective controls. At the end of the same period, the calcium-cyclamate sodium-saccharin combination samples had also developed a detectable difference. The control samples, sweetened with sucrose, had a 50% longer storage life at 85° F before a detectable difference occurred. The storage life of 9 weeks with the sucrose sweetened grapefruit samples was still somewhat less than normally expected with foam-mat dried unsweetened grapefruit crystals which may run as high as 12 weeks or longer (3).

The equivalent flavoring levels of the other grapefruit flavoring agents studied and storage times at 85° F for development of detectable differences are shown in Table 3. In comparison to "locked-in" oil added at 0.005% level, it was found that equivalent level of peel oil was 0.0076% and of essence oil was 0.0078%. The Polak commercial grapefruit flavoring was found equivalent in grapefruit flavor to the control when it was added to grapefruit crystals at a level of 0.2%. When these samples were compared in storage at 85° F, none was found to have an equivalent storage stability to that of the control. Whereas a standard sample with "locked-in" oil lasted for 12 weeks before a detectable change occurred, the samples flavored with cold-pressed peel oil, or with essence oil, had developed detectable differences at six weeks. The sample with commercial grapefruit flavoring developed a detectable difference at the end of five weeks. Thus, it would appear that

Table 1. Equivalent sweetness of three agents in grapefruit crystals using triangle taste tests.

Sweetener	Level used %	Results Right-Wrong
Sucrose	27	Control
Calcium cyclamate	0.3	10-14*
Ca Cyclamate/Na Saccharin (110/1)	0.2	12-12*

* No significant difference when compared to control.

Table 2. Storage tests on sweetened grapefruit crystals at 85°F. using triangle taste tests.

Sweetener	Weeks for detectable difference
Ca Cyclamate	6
Ca Cyclamate/Na Saccharin	6
Sucrose	9

Table 3. Flavoring agents for foam-mat dried grapefruit crystals and storage tests determined by taste.

Agent	Equivalent levels in crystals %	Type test	Weeks @ 85°F. for detectable difference
Peel oil	0.0076	Triangle	6
Essence oil	0.0078	Triangle	6
Comm'l. Grpft. flavor	0.20	Pair	5
"Locked-in" oil	0.005	Triangle	12*

* Food Technol. 20(4) 177-78 (1965).

the "locked-in" peel oil enhances the overall storage stability of these grapefruit crystals.

In the study of the relative acceptability of different oil levels in grapefruit crystals, several interesting observations were made. The results of this study are shown in Table 4, and indicate that among relatively inexperienced tasters a high oil level is preferred. The low oil level (0.0075%) received an overall score of 5.06 on a Scale of 1 to 10. The medium oil level (0.010%) received an overall score of 5.56 and the high level (0.012%) received an overall score of 5.62. Although there is no significant difference between the scores of the medium and the high levels, the difference between the lower level, (0.075%) and the other two levels, is significant at the 95% confidence level. A detailed statistical analysis of the data was performed by Statistical Reporting Service, Washington, D. C., and copies of this are available from the authors.

Table 4. Mean preference scores for three levels of "locked-in" oil added to foam-mat dried grapefruit crystals.

Oil level %	Overall score	Users score	Non-users score
.0075	5.06	5.24	4.89
.010	5.56	6.07	5.05
.012	5.62	5.70	5.54

Scores are mean values of 3 replications of 38 tasters.

When these data were separated between users and non-users, another interesting observation was made. It was found that the middle oil level, 0.010% was preferred by users whereas the higher oil level 0.012% was preferred by non-users. In every case preferred levels were higher than the 0.005% which had previously been used routinely with foam-mat dried grapefruit crystals in our laboratory. The fact that the non-users gave the higher score to the higher oil level might also be an indication that foam-mat dried grapefruit crystals in order to appeal ton the non-users' market, would be more effective at higher oil levels.

Freeze-dried single strength grapefruit juice was also tested as a flavoring additive when added to foam-mat dried grapefruit crystals. A sample of freeze-dried grapefruit juice with very high initial quality was prepared and added at levels of 5, 10, 20, and 30% to foam-mat dried crystals. It was found that no significant difference was apparent when 5% freeze dried material was used. The sample with freeze-dried grapefruit was significantly different and preferred over the control when it was added at the 10% level and higher. Storage data on grapefruit samples with these additives has not been completed to date.

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