(86°F) contained Voges-Proskauer (VP) reactants. Over 1 ppm of diacetyl was found in one sample analyzed by the distillation procedure. Oil analyzed by gas chromatography showed alpha-terpineol was produced which increased throughout the test period.

Four test organisms were isolated from orange serum agar pour plates. All grew rapidly when inoculated into sterile orange oil emulsion. One did not produce alpha-terpineol.

Production of alpha-terpineol in orange oil is believed to be a result of bacterial growth, and not a chemical and/or oxidation process.

Good sanitary practices must be maintained in order to control contamination during the orange peel oil recovery operation.

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RELATION OF PRESSURE AND MOISTURE FOR DENSIFYING FOAM-MAT DRIED ORANGE AND GRAPEFRUIT JUICE CRYSTALS[1]

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ABSTRACT

Previous studies have indicated that foam-mat dried citrus juices require densification for improved reconstitution properties. This may be achieved by application of pressure. A study was made of the relation between pressure applied, moisture content of the citrus crystals, and the degree of densification achieved in orange and grapefruit foam-mat dried crystals. Samples were prepared which varied in moisture content from 0.9 to 1.6% and these were subjected to pressures of 3,800 to 19,100 psi (pounds per square in.) by a ram driven hydraulic press. This treatment formed small, firm, citrus "discs" which were then tested in a shear press to determine the amount of shear required to break them. This shear strength was then taken as an index of the degree of firmness, or densification achieved. Some differences were found in the behavior of orange and grapefruit samples. Orange crystals generally increased in firmness of the discs with pressure at all moisture levels. With grapefruit samples, however, there was a definite peak in degree of densification at about 13,400 psi. At pressures of 15,300 and 19,100 psi, the firmness of the formed discs dropped sharply. With orange crystals the degree of densication at a given pressure generally increased with increased moisture, but with grapefruit optimum moisture content was 1.3%.

INTRODUCTION

Increasing interest is being shown in methods for dehydration of citrus juices. Most methods presently under consideration for this purpose produce a very low density dried material with a coarse structure. This is particularly true of the foam-mat drying process which has been under study at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven. This low bulk density has been found impractical for commercial purposes and methods have been investi-

[1]Cooperative research of the Florida Citrus Commission, the Western and Southern Utilization Research and Development Divisions, Agricultural Research Service, U. S. Department of Agriculture.
References to specific commercial products do not constitute endorsement.

gated for increasing the bulk density by a densication process in which pressure is applied to the porous material, (Berry, et al., 1965 and 1967).

Although densification methods have been developed, the degree of densification achieved under given conditions varies. Past studies indicated moisture content of the foam-mat dried powder was related to its behavior during densification. No data were available on this relationship.

In order to provide information which might establish guidelines for commercial application of the densification process to citrus crystals a special study was undertaken. In this study foam-mat dried orange and grapefruit crystals containing different amounts of moisture were prepared. These were subjected to different pressures on a hydraulic press, forming small discs. Measurements were then made of the thickness of the discs formed and of the shear strength of the discs as determined by amount of shear required to break them. The results of that study are the subject of this report.

Experimental

Citrus Crystals

Orange and grapefruit crystals used in this study were prepared on a crater type foam-mat drier by the method described by Berry et al (1967). All samples contained 0.45% methyl cellulose as a foaming additive. They were prepared from 50° Brix commercial grapefruit or orange concentrates and all samples had a high initial quality. For these experiments no flavor additives were used. Foam-mat dried citrus juice directly removed from the drier was ground to pass through 20 on 60 mesh screen (U. S. sieve size). This material was used as the starting material in all experiments. It had an initial bulk density of .25 g/ml, and an initial moisture level of 1%. The moisture content was adjusted to levels desired for experimental studies and samples were sealed in air in tin cans at atmospheric pressure.

Moisture Levels

In order to adjust to exact desired levels of moisture in the samples for densification experiments the following procedure was used. After determining the initial moisture content 100 g of foam-mat dried citrus juice crystals were placed in a No. 2 (307 x 409) tin can. Onto a small piece of cotton gauze was absorbed the amount of water required to convert the sample to the desired moisture level and this gauze was taped to the inside of a can lid. A can of citrus crystals was then sealed with the lid and placed in a 70°F room for 3-5 days. At the end of this time the citrus juice crystals were mixed thoroughly and a moisture determination was made by a modification of the Karl Fischer method as described by McComb and Wright (1954). Samples in the range of 0.9 to 1.6% moisture content were prepared after the desired level was reached samples were then sealed and stored at 50°F until used.

Citrus Discs

Experimental discs of different samples of orange and grapefruit crystals were prepared. Two g of the undensified material were placed in a one-in. diameter cylinder on a Carver hydraulic press and a ram was pressed into the cylinder to the desired pressure and held for five sec. Forces from 3,000 to 15,000 lbs were applied to the ram (3,800 to 19,100 psi, on the citrus discs, respectively). The pressure on the discs was determined from the force applied by dividing total force on the ram by the area of the ram end, i.e., psi $= \dfrac{\text{Force on ram (lbs)}}{\text{Area of ram (in.}^2\text{)}} = \dfrac{F}{\pi r^2}$ for a one-in. ram $= \dfrac{F}{\pi/4}$ psi. All values are reported in psi on the discs and not in lbs ram force. The thickness of the discs thus formed were measured with a micrometer caliper as an index of the degree of compressibility achieved.

Shear Strength Determination

The shear strength of the discs was determined by using an Allo-Kramer Shear Press, Model SP-12 to measure the amount of force required under standard conditions to break the discs. The Shear Press consists of a device for pressing a number of flat blades (blade element) through the test material in a slotted container (shear cell). The blade assembly is attached to a large "proving" ring which compresses as pressure is applied on the blades. This ring in turn compresses the sensitive point of a transducer which translates the pressure to a recorder. For these studies a standard "300 lb" proving ring was used. In order to check the calibration of the instrument before each determination a shear value was determined on a number of small rectangles of paper as a control measure. This was repeated also after each adjustment of the instrument. When necessary the instrument was recalibrated prior to determination of experimental samples. For sample determinations three discs were placed in the sample container and the blade was lowered at a speed setting of three on the instrument (5.33 in./min). The recorder was adjusted to give maximum accuracy on each determination as required. At least three determinations were made on discs from each different sample at each different setting and the average values were tabulated.

Results and Discussion

Generally, two methods were used for evaluating results of the compression of foam-mat dried crystals. One consisted of measuring the thickness of the discs formed and the other consisted of measuring the shear required to break the discs. The first method is obviously a direct measure of the increase in bulk density and the second is actually a measure of the shear strength. This strength factor is of value since it has been observed in previous studies to be related to degree of formation of "fines" during grinding of densified plates or particles of foam-mat dried citrus (Berry, et al., (1967)). Generally, the firmer the material the less "fines" formed during grinding.

The thickness of the formed discs and the shear required to break the discs made from orange crystals of different moisture contents using 3,800 to 19,100 psi are shown in Table 1. Generally, the disc thickness decreased, that is, the discs were compressed more completely and the discs required more shear to break them as the applied pressure increased up to 15,300 psi. Beyond that there was a slight decrease in shear strength at 1.6% moisture and a sharp decrease at 1.3% moisture. In most cases with discs formed at a given pressure the shear strength was greater as moisture content increased. There was some variability in those formed at the lowest and highest pressures. The compressibility of the powder as shown by disc thickness also followed this trend. At any given pressure the discs formed from orange powder with higher moisture contents were more thoroughly compressed (thinner) than those where moisture was lower. The measured values for shear strength varied from a low of 300 lbs for a 1.3%

Table 1. Thickness and shear strength of discs formed from orange crystals of different moisture contents at different applied pressures.

Disc-forming pressure ($psi \times 10^2$)	Thickness ($in. \times 10^{-3}$)			Shear strength (Shear to break disc, lbs)		
	Moisture content			Moisture content		
	1%	1.3%	1.6%	1%	1.3%	1.6%
38	148	144	139	388	300	680
76	131	127	115	775	925	1,400
115	126	114	108	880	1,338	1,540
134	123	120	108	1,070	1,430	1,600
153	119	110	106	1,200	1,610	1,740
191	116	112	112	1,310	1,270	1,700

All are values averages of three determinations; Shear $\pm$ 70 lbs; thickness $\pm$ 3×10^{-3} in.

moisture content to a high of 1,740 lbs for a disc of 1.6% moisture content. The disc thickness varied from 0.1480 in. witih a 1% moisture content disc formed at 3.280 psi to 0.1065 in. disc at 1.6% moisture formed with 15,290 psi.

Foam-mat dried grapefruit crystals were given similar treatments at different pressures and moisture levels and results are given in Table 2. Generally, the disc thickness became less with increasing applied pressure in most samples. However, there were a few variations, especially at higher disc-forming pressures and the shear required to break the discs varied considerably. The 1.3% moisture discs, formed using 3,800 psi were considerably harder than the 0.9% moisture discs, or the 1.6% moisture discs, formed under similar conditions. Shear required to break these discs was 660 lbs as compared to 345 and 355 lbs, respectively.

At 7,600 and 11,500 psi disc-forming pressure the 1.3% moisture sample produced the discs with highest shear strength followed by the 0.9% moisture sample and the 1.6% moisture sample in that order. From 11,500 to 15,300 psi applied force the 1.6% moisture sample produced stronger discs than did the 0.9% moisture sample, but the 1.3% moisture samples were still *firmest in this* range. Thus, at all pressures studied with grapefruit the optimum moisture was 1.3%. Grapefruit disc thicknesses varied from a high of 0.147 in. at 0.9% moisture to a low of 0.1040 in. for 1.6% moisture. The shear to break the discs varied from a low of 345 lbs for 0.9% moisture samples formed at the lowest pressure to a high of 2,325 lbs in the 1.3% moisture sample formed using 13,400 psi applied pressure. The general range of values of disc thickness and shear required to break discs was about the same with grapefruit as with orange. That is, neither was particularly more compressible than the other.

This may be further substantiated by consideration of the relationship between the disc-producing pressure and the shear stress. Shear stress is the shear strength divided by the shear area, (i. e., in this case, the thickness of the discs). When these values were considered, the data indicated little difference between orange and grapefruit in disc-producing pressure at which maximum shear stress was observed. Generally, with both orange and grapefruit, the disc-producing pressures of 13 to 16 thousand psi resulted in discs having maximum shear stress.

Table 2. Thickness and shear strength of discs formed from grapefruit crystals of different moisture contents at different applied pressures.

Disc-forming pressure ($psi \times 10^2$)	Thickness ($in. \times 10^{-3}$) Moisture content			Shear strength (Shear to break disc, lbs) Moisture content		
	.9%	1.3%	1.6%	.9%	1.3%	1.6%
38	147	140	143	345	660	355
76	126	125	128	845	930	775
115	119	116	120	1,110	1,325	1,025
134	111	108	113	1,400	2,325	1,590
153	114	109	104	1,225	1,490	1,580
191	114	106	106	965	1,375	1,525

All values are averages of three determinations: Shear $\pm$ 70 lbs, thickness $\pm$ 3×10^{-3} in.

The moisture level of 1.3% found optimum in these studies is in agreement with results from actual densification observations on a pilot plant scale. The optimum densification pressures observed here are only about half those observed effective in pilot runs, however. In the pilot runs the foam-mat crystals are fed into the nip of nine-in. diameter heavy steel rolls turning about 16 rpm and removed by a doctor blade. The differences between pilot runs and laboratory runs with respect to pressure required are probably due to differences between a static system (as in the present study) and a dynamic system, (as in the pilot plant studies). In the statis system, the sample is subjected to pressure over a relatively long time (5 sec) whereas in the dynamic system the pressure is only applied to the sample for a fraction of a sec. This probably accounts for the differences in required pressures in the two systems. The studies with the static system have shown that it is possible to use pressures which are too high for optimum densification, and this is in agreement with observations in the dynamic system. Thus, although the values are not directly comparable, the same principle appears applicable in both systems.

A coincidental factor in this study was the suggestion of a prospective new form for a citrus product. During this study, several investigators began eating some of the formed discs and found that they were very tasty. The amount of solids equivalent to one ounce of juice was also found to compress nicely into a bite-sized disc of good proportions for eating. Several of these have been circulated to interested parties for samples and have been favorably received. They could probably be coated with chocolate or other material, or dusted with starch to reduce hygroscopicity and eaten like candy, or perhaps blended with some ingredient to make them fracture and fragment easily and then be used to make juice. They might also be used as a compact chewable form of citrus for space flights and the like.

In summary, the conditions for densification of orange and grapefruit foam-mat dried crystals have been studied. Generally, the lower the moisture content the higher the pressure required for densification of orange, up to 15,300. For grapefrut 13,380 psi was optimum with each moisture level studied, and 1.3% was the optimum moisture level. A coincidental result of the study was the observation that these discs might provide a new product form for citrus.

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AN IMPROVED METHOD FOR MEASUREMENT OF PULP CONTENT OF CITRUS JUICES[1]

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Abstract

Modifications were made to the currently used standard centrifugal method for determining sinking pulp in reconstituted frozen concentrated orange juice as described in the Florida Citrus Commission Regulation 105-1.19. Seventy-four samples of commercial frozen concentrated orange juice, packed during the 1965-66 and 1966-67 citrus seasons, were tested by both the standard and modified methods. The samples were chosen from each season's packs to provide primarily a wide range of apparent and relative serum viscosities. Comparison of data indicated that precision or "repeatability", using the modified method, was better than that when the standard method was used. In general, precision was better with low viscosity juices regardless of the method used.

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