Pistacia oil, the collection of more data in a wide range of percentage in FFA was needed. To accomplish this, samples of pistacio oil examined were mixed in a manner to produce a variety of new samples covering the range of 0–20% in FFA content.

For all the new samples the FFA content and the laboratory refining loss according to the chromatographic method were determined. Three or four specimens were examined for each sample and the mean value was taken. Table III shows that this value is increased by increasing percentage of FFA.

A plot of mean values of the refining loss against percentage of FFA gives a straight line. The slope and the intercept of this line, calculated by the method of least squares, were found to be, respectively, $b = 1.015$ and $a = 0.81$. The s.d. of b and a were found to be $S_b = \pm 0.029$ and $S_a = \pm 0.30$.

It is evident that even within two standard deviations, *a* exists and therefore— as expected from the theory—a refining loss exists even when the FFA content equals to zero.

The correlation coefficient of the straight line obtained was found to be $r = 0.99$ giving a very good criterion of linearity.

Consequently, for the average relationship between refining loss determined by the chromatographic method and FFA content of Pistacia oil, the following expression may be proposed:

percentage refining loss = 0.81 + 1.015 (percentage FFA)

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The Expansion and Extraction of Rice Bran

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Abstract

Expansion of rice bran as a pretreatment for solvent extraction was studied. It was found that the expanded bran showed no rise in free fatty acid (FFA) even when stored at room temp, in open containers, for a period of three months, and a slight rise after one year storage; and that the bran was agglomerated into large particles which eliminated the "fines" and channeling problems characteristic of rice bran; and that the retention time for good extraction was on the order of 45 min; and that the percolation rate for a four ft depth of expanded bran was on the order of 35 gpm/ft^2.

Introduction

THE PROBLEMS ENCOUNTERED in the solvent extraction of rice bran are well known and widely reported and have rendered the usual type extractors, the total submergence columns and percolation units, practically useless in handling rice bran. The problems with rice bran are associated with its finely granulated nature and its tendency towards rapid conversion of the rice oil into FFA. Fines cause problems in the clarification of miscella (4,10), the condensation of the fines-ladened vapor from the desolventizers (4) and channeling within the baskets (4); they have become so troublesome as to force some operators to run their extractors far below capacity (10).

The rapid conversion of rice oil into FFA is due to the action of a lipolytic enzyme which is activated during the milling operation (7) and, to a minor extent, the action of bacteria and molds in the presence of air and moisture (6). The FFA level increases rapidly (6,7,17) reaching a level of 50–70% in only 90 days storage time (7). A variety of methods to inhibit the rise of FFA have been reported, some of them by chemical methods (7,17), and others by heat sterilization of the bran and storage under arid conditions (1,2,4,7,13).

When heat sterilzation treatment is given in the presence of a moisture level sufficient to cook the material, the additional benefit of fines agglomeration is obtained (4). This development of cooking rice bran to eliminate the fines and retard the FFA formation, which are the two most vexing problems concerning the processing of rice bran, would render rice bran as easily extracted as any other well prepared oil seed (1,2,4,12,13).

The deactivation of the lypolytic enzyme and the sterilization of the bran due to the temp of cooking, enables the treated bran to be stored for long periods of time with no significant increase in FFA. Cooking also releases the oil (4) similar to the effect of cooking upon other oleaginous materials. The fines agglomeration, which is due to the gelatinization of carbohydrates and proteins contained in the bran (2,4,12,13), helps to increase the percolation rate of the treated material as well as eliminate the problems encountered in the miscella clarifiers and meal desolventizers.

Much of the work with rice bran has been done by Southern Regional Research Laboratory, who published their results in a series of journal articles (2–4, 16).

Experimental Procedures

Rice Bran. The rice bran used for this project was obtained from the Blue Ribbon Rice Mills Inc.,

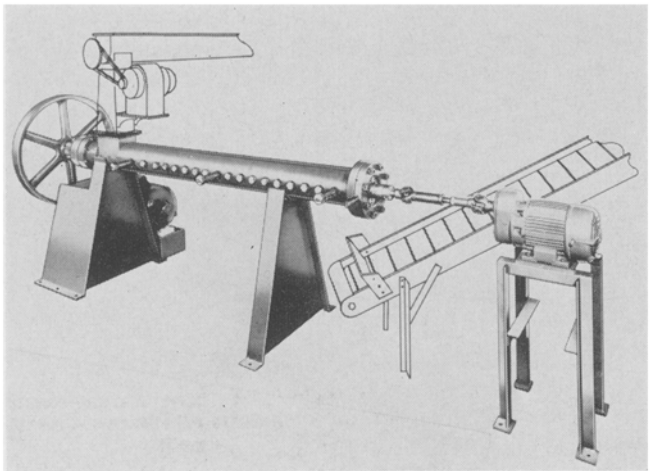

FIG. 1. Exterior view of a typical Anderson horizontal grain expander.

Houston, Texas. A preliminary series of tests was made to evaluate the cooking and agglomerating effects of expansion with no particular attention to the FFA content in the bran. Then a second series of tests was made, paying particular attention to the FFA content as well as confirming the cooking and agglomerating data from the first tests. The bran for the second test was shipped air express and was expanded two days after milling, at which time the FFA level had reached approx 7%.

Description and Operation of the Expander. The V. D. Anderson Expander consists of a cylindrical cooking vessel equipped with a rapidly rotating screw shaft having interrupted flights and provided with a number of small orifices on the discharge end through which the cooked material is extruded.

Sometimes the Expander barrel is steam jacketed, but generally the steam jackets are not required, and, on most Expanders, are omitted altogether. In such cases, the heat of cooking is supplied by the shaft friction against the material to be expanded and by injected steam. Sometimes cereal grains are expanded without addition of moisture; but usually, especially with materials other than cereal grains, moisture is added to the material as water or sparge steam, or both, through especially designed breaker bolts which serve as injectors. The Expander is shown in Figure 1.

The pilot-scale Expander used for this project has a cooking chamber, or barrel, of 4.5 in. ID and 4-ft length. A single die orifice with a circular opening of $\frac{5}{16}$ in. diam was used in the flat discharge plate. The shaft speed was 216 rpm powered by a 25 HP motor. The steam injector was located ca. 1 ft from the discharge end of the barrel; the water injector ca. 3 ft from the discharge end.

The procedure for a typical run would consist of feeding the rice bran as received, into the feed hopper of the Expander directly onto the first, or feed worm of the rotating shaft. The material is conveyed for ca. 6 in., then it comes into contact with injected water. Then it is conveyed through the barrel for an additional distance of 1.5 ft, where it comes into contact with injected sparge steam, and the moisture of the material is finally adjusted to the proper level for expanding. For rice bran the proper moisture level is approx 25% by wt of wetted sample. Then the wetted material is pressed by the rapidly rotating shaft against the stationary die plate where its flow out of the barrel is restricted by the small die orifice, thereby causing the material to be placed under a high pressure. The material is also heated, either by the shaft friction alone, or a combination of shaft friction and injected steam until the temp of the material reaches 250–325F. While inside the Expander, the material is blended by the rapidly rotating shaft and, due to the heat present, is cooked to such an extent that enzymes are inactivated and raw starch gelatinized. The water in the material is kept in the liquid state by the high pressure maintained within the Expander. Immediately upon discharge through the die, the sudden vaporization of the superheated water causes the material to be blown full of internal pores and thereby swelled to many times its extruded size. Usually the bulk density of a material is decreased by expansion, but with rice bran and similar materials it is slightly increased.

The position and number of water and steam injectors, the size and number of die orifices and the shaft speed can be varied to provide for adjusting the operating conditions for each particular material being treated. The expanded product can lose up to ca. one-fourth its moisture content through flash cooling, or, it can easily be dried to a specific moisture content in an apron dryer.

During the expander runs, a list of data was compiled showing the conditions used for each text sample.

Glassware Percolation Rate Tester. The percolation rates of the samples of expanded rice bran were determined in a pyrex glass tube 1.65 in. ID, 4 ft long and capped at the bottom with a 14-mesh stainless steel screen, having a cross-sectional area of 2.14 in.^{2} The expanded rice bran was poured into the column to a

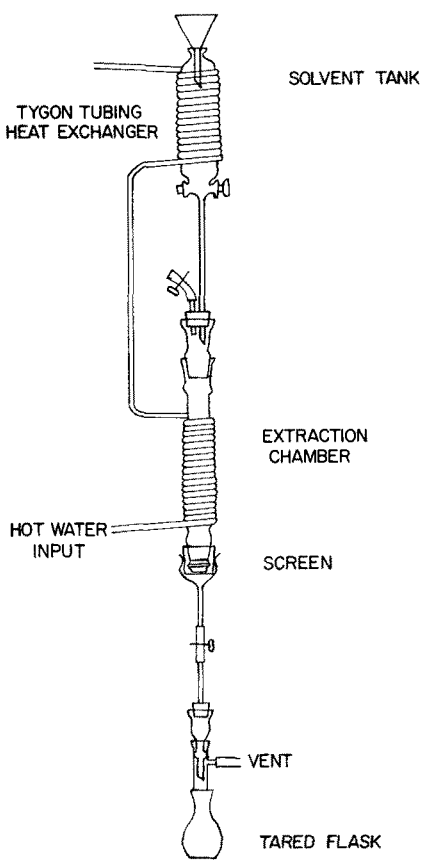

FIG. 2. Glassware extraction apparatus.

TABLE I

Expander Data

	Crumb (1 pass)		Rope (2 passes)
	(Vertical)	(Horizontal)	(horizontal)
Feed conditions			
Bran moisture content %	8.3	9.0	20
lb/hr Bran input rate	66.7	128	1890
lb/hr Water input rate	14.9	22	10
lb/hr Steam input rate	0	10	140
Expanding conditions			
Moisture content %	23.4	27	27
Internal temp °F	320	240	
Temp after discharge °F	250	180	
lb/hr Throughput rate	81.6	160	2040
Horse power consumed	12	10	22.5
Conditions of product			
% Moisture after discharge	12.7	23	26
Bulk density lb/ft³	20	25	25.5
Coefficient of expansion	1.24	1.24	1.28

depth of 3.5 ft, with periodical rapping of the column against the floor to settle the bran down into a compacted bed in order to insure that the column is packed as tightly, or more tightly, as a basket in a horizontal basket extractor. Then normal hexane was percolated through the column, collected in a graduate cylinder and the following data obtained: the penetration rate of the liquid through the bed was recorded in terms of average in./min travel for the particular bed depth tested; the percolation rate of liquor passing through the column under flood conditions was measured in terms of ml/min/2.14 in.^{2} screen area and converted to gpm/ft^2; and finally the drainage time required to free drain the column after loss of liquid head above the bran was recorded. The miscella was recycled through the column and another set of data obtained. Generally a higher percolation rate was obtained for the first trail than for the second trial due to the filtering from the miscella of fines which help to seal the bed and to the additional bedding of the bran in the column.

Extraction Rate Test Apparatus. The extraction rates were determined in a pyrex glass tube 7/8 in. ID, 8 in. long, with a stainless steel screen secured to the bottom by means of a bored rubber stopper. The glass column was wrapped with Tygon plastic tubing through which hot water was passed in order to maintain an extraction temp, within the column of 140–150F. A separatory funnel, similarly heated, filled with clean hexane, was placed above the extraction column so that the hexane could be metered through a two-hole rubber stopper into the extraction column. The other hole in the two-hole rubber stopper was equipped, by means of tubing and a pinch clamp, to vent the extraction column whenever necessary. The miscella passing through the extraction column is allowed to drain from the separatory funnel, into the bran sample contained in the glass tube, then past the stainless steel screen, through a glass stopcock and past a permanently opened air vent into a tared flat-bottomed boiling flask of 250 ml capacity. Figure 2 shows drawing of the glassware extraction apparatus.

The extraction rates were determined according to two procedures. Both procedures have been checked numerous times against countercurrent extraction operations and have proved to be reliable. The first consisted in charging the column with bran, then allowing the hexane to leach through the bran for a predetermined retention time while keeping the column flooded and adjusting the miscella flow with the stop-cock below the column; then the column is allowed to drain free of excess hexane and a representative sample of the extracted bran is obtained to be analyzed for residual oil. The column is then recharged with the bal-

TABLE II

Absorption of Moisture by the Extracted Expanded Rice Bran

Days exposed	% H_2O in bran
0	2.2
1	4.1
2	6.4
4	....
7	7.4
10	7.5
14	....
16	7.8
30	6.8
42	10.3
56	8.6
107	9.1

ance of the extracted bran, and the extraction cycle is continued until the bran supply is exhausted. The residual oil figures obtained are then plotted against extraction time as is recorded in the results.

The second procedure consists of charging the column with an accurately-weighed sample of bran at a known moisture content, and allowing hexane to leach through and be collected along with the extracted oil in tared boiling flasks. At the end of five min the flask is replaced with a clean tared one and the extraction is continued while the miscella in the first flask is dried down and the oil weighed. The flasks are continuously replaced at varying time intervals until a total retention time of one hr has been attained, at which time the extracted bran is removed from the glass column and analyzed for residual oil.

The wt of oil removed with each tared flask are used, along with other data from the test, in order to prepare a material balance for each time interval during which a flask of miscella was removed from the extraction apparatus. From the material balance, data is obtained showing the relationship of residual oil content to extraction time.

Determining the Degree of Rice Bran Sterilization. All of the rice bran after expansion, as well as the feed sample, had a FFA content of approx 7%. Samples of expanded rice bran, representing each of seven tests with the expander, were divided into four portions for testing purposes. The portions were: 1) expanded bran as is (no additional drying), stored at room temp (60–80F); 2) expanded bran, dried to 6–9% moisture content and stored at room temp; 3) expanded bran as is, stored in the refrigerator at a temp of 41F; and 4) expanded bran, dried and stored in the refrigerator. A sample of expanded bran was over-dried for four hr at a temp of 212F to a moisture content of 1.2%. This was then portioned into two samples, one stored at room temp, the other in the refrigerator. A quantity of the feed, or raw bran, was

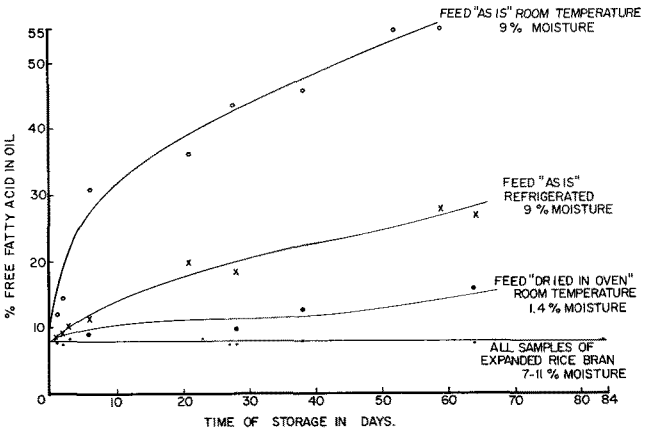

FIG. 3.

TABLE III

Sieve Analysis of Raw Bran and Expanded Crumb

U.S. series sieve no.	% Raw bran on sieve	% Expanded crumb on sieve
4	.....	28.2
5	.....	5.7
6	.... .	7.6
8	.... .	13.1
10	....	10.4
14	0.8	
24	23.5	22.3
40	41.0	8.5
60	13.7	2.8
100	13.2	.... .
200	6.2	....
Pan	1.1	1.4

divided into three samples: 1) feed, as is, stored at room temp; 2) feed, as is, stored in the refrigerator; 3) feed, dried in an oven for three hr at a temp of 212F to a moisture content of 1.4%, and stored at room temp. Each sample was placed in a glass jar with the lid laid loosely over the top to keep out foreign matter. The lids were removed from the jars, and the contents stirred daily, to insure adequate contact of the sample with the air.

The FFA content of the oil in each sample was determined periodically for a period of one year, the determination being made according to AOCS Methods Aa 6-38 and Ca 52-40, both methods giving similar results. The oven-dried samples were prepared as controls representing the conventional method of heating to sterilize the bran. A thorough study of this method of treatment is described by Loeb (7).

Results

The Expander Operation. Two trial runs in two different models of the Expander were conducted, one in the Model Horizontal Expander as illustrated in Figure 1; the other, a preliminary test, in an earlier version of the Expander. Both Expanders agglomerated the rice bran into discrete particles which had good percolation and extraction rates. No study of the lipolytic enzyme was made during the preliminary test since the rice bran used was not fresh from the mill.

Part I. Data From the Preliminary Run in the Vertically Arranged Machine

Conditions of Expander. Expander barrel approx two ft long with three in. ID; shaft turning at 212 rpm powered by 20 hp motor; water injected into feed trough before entry into Expander; no sparge steam injection, one die opening in discharge plate of size 0.25 in. and square shape, giving an orifice of 1/16 sq in. area and one-in. perimeter; 50 psig steam pressure on barrel jackets.

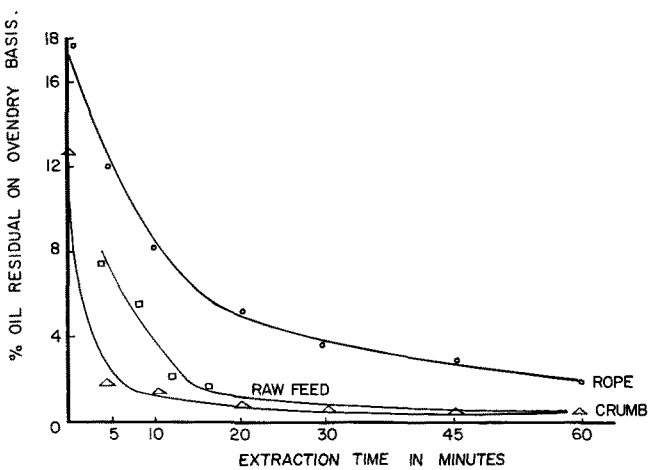

FIG. 4.

Conditions of Expansion. The rice bran was wetted in the feed trough to 23.4% moisture content and was conveyed into the Expander where it was heated to 320F before discharge whereupon the temp dropped immediately to 250F and then slowly to room temp at which time the moisture content had dropped to 12.7%. The oil content of the uncooked rice bran was 16.2% at 8.3% H_2O; that of the expanded bran was 16.7% at 5.0% H_2O showing no alteration of oil yields due to expansion. The bulk density of the expanded rice bran when compared to the feed showed an increase in bulk density of approx 24%. The bran was expanded into pellets, porous in nature and approx 1 in. long by 0.25 in. diam.

Part II. Data From the Run in the Model Horizontal Expander

Conditions of Expander. Expander barrel 4 ft long with 4.5 in ID; shaft turning at 216 rpm powered by a 25 hp motor, water injected into feed within the barrel; and sparge steam injected just before discharge. One die opening in discharge plate of size 5/16 in. circular opening. Jacket steam was not used.

Conditions of Expansion. The rice bran was fed without pretreatment, directly to the Expander, and was treated under varying conditions similar to those described for the preliminary run. The second run was broken down into five distinct tests with raw bran and two tests with recycled bran. The data from the Horizontal Expander runs are summarized in Table I. Tests 1–5 of this run produced a soft, loosely-held product which crumbled up easily into a granular state. Tests 6 and 7 with recycle bran, produced a rope-like product which did not crumble. After it was noted that all of the expanded bran showed no increase in FFA level, the samples of each tests were combined into two samples: one of crumb, and one of rope-like product. The extraction tests were performed on these two composite samples.

The coefficient of expansion is obtained by comparing the bulk densities of the feed and the product (F/P = C of E) when each sample is ground to the same particle size and the calculations are based upon a no-moisture basis. The coefficient of E is similar for both Expander runs indicating a rise in density, after expansion, of 24–28%.

Laboratory Tests

Part I. Inactivation of Lipolytic Enzymes

No increase in FFA content of the oils in any of the expanded samples were noted during an 84-day storage period. A spot test after one year of storage showed a FFA content of 7.4%, and a spot test after 1.5 years of storage showed a FFA content of 8.0%. This is from bran which had 7.0% FFA in the oil before it was expanded. See Figure 3 for the plotted FFA curves.

Part II. Absorption of Moisture by the Solvent Extracted Meal

A sample of expanded rice bran in the crumb form was solvent extracted to 1.27% residual oil content, and was placed in an open dish for a period of 107 days. This exposure period included the summer months to insure that the meal would be exposed to a humid atmosphere. The results are tabulated in Table II.

Part III. Extraction Tests

See Table III for a comparison of the sieve analysis of the raw feed, and the expanded crumb; Table IV for the percolation data; Figure 4 for the plotted extraction curves. Although the expanded bran is compared directly to raw bran the work of Gastrock et al. (2,4,15) with filtration-extraction can also serve as a basis for comparison.

Discussion

Before a material can be expanded, it should be cooked. The cooking is obtained within the Expander barrel, and the expansion is obtained immediately upon discharge of the material through the die orifices. As a matter of procedure, pilot plant tests generally cover a range of cooking conditions to determine which conditions of cook are required to pretreat a material before expansion can occur. This was done with rice bran and the proper conditions are recorded in the data. Actually, there is a wide range of conditions adequate for the preparation of rice bran for solvent extraction through use of the Expander, perhaps wider than the range recorded. When the cooking conditions are relatively mild, the expanded product appears in an easily crumbled form. When the cooking conditions are more severe, the product occurs in a continuous rope-like form which can be cut into pieces of any desired length by means of a rotary knife blade attached so that it passes over the die orifices.

TABLE IV

Percolation Data

Sample	Penetration rate	Percolation rate	Drain time	M & V of marc
Expanded rope-like cake	241 in./min	140 gpm/ft²	0.3 min	25%
Expanded crumb	98.4 in./min	35 gpm/ft²	1.6 min	30%

The degree of expansion in both forms was minimal, which was to be expected, due to the low starch content in rice bran. The rope form is usually more desirable in an expanded product, but the crumb form was found to be superior to the rope form because it is more easily extracted in a solvent extractor.

The essential objectives in preparing rice bran for solvent extraction are 1) to prevent any rise in the FFA content; 2) to agglomerate the particles of bran together in order to reduce or eliminate the fines portion and to prevent channeling in the extractor baskets; and 3) to obtain a percolation rate in the extractor baskets of not less than 5 gpm/ft² in order to insure adequate mass transfer rates of oil from the bran into the solvent.

These objectives are fulfilled by expansion in both forms of finished product. But since the crumb requires a much shorter retention time for solvent extraction, due to its smaller particle size, it is preferred over the rope-like product. The retention time for the crumb is very smiliar to that required by flaked soybean. The extracted meal, moreover, is not as hydroscopic as extracted meal from raw rice bran; and the rice bran, after expansion and drying, can be stored under the usual conditions for long periods of time before it need be extracted for the rice oil .

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Assistance of G. A. Soltis in obtaining some of the extraction data; help from R. P. Gortz in obtaining the FFA data; aid from T. E. Pahoundis and Thomas Guilliford in the Expander operation; and aid from Jean Mehalik in preparing the manuscript.

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Determination of Weight of Bulk Oil Shipments

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Abstract

The following is a brief consideration of the methods used in the determination of wt of bulk shipments of vegetable and animal fats and oils, with particular reference to tallow. Sources of error are noted, and the techniques involved in accurate specific gravity determinations are discussed.

Introduction

AS MOST BUYERS AND SELLERS of vegetable and animal fats and oils are aware, shipments by vessel are carried largely in bulk. Since buyer, seller, vessel owner and insurance agent all require accurate measurements or determinations of wt at both point of origin and point of discharge, we believe a brief discussion of the techniques involved would be of interest to the industry in general. This firm has been actively involved in the surveying, inspection and chemical work connected with bulk vegetable and animal oil shipments ever since the first delivery in bulk was made at San Francisco some 50 years ago. During this time we have witnessed vast changes in methods and practices and have had the opportunity of learning a great deal, not only from our own experiences, but from reports by other surveyors in many parts of the world. To the best of our knowl-