

Rice Bran Oil. IV. Storage of the Bran as It Affects Hydrolysis of the Oil¹

J. R. LOEB, N. J. MORRIS, and F. G. DOLLEAR, Southern Regional Research Laboratory,² New Orleans 19, Louisiana

Introduction

RICE BRAN, the term applied to the coating removed from brown rice during milling, is composed of the seed coat, the major part of the germ, and most of the outer layer of the kernel together with some broken kernels. The bran which constitutes about 8.5% of the whole grain is highly nutritious owing to its content of fat, protein, and vitamins (6). Because of the nutritional value of rice bran it has been used as a feed for poultry and livestock. However in addition to its value as a feedstuff, rice bran constitutes a potential source of oil. The bran contains from 14-18% of an oil with properties similar to that of cottonseed oil (4). Moreover the removal of the oil from the bran should not impair its value as a stock feed since the protein and vitamin contents should be correspondingly increased by removal of the oil and it should be more stable with respect to the oxidative rancidity which is often a serious problem with this mill feed.

A major difficulty of rice bran as a raw material for the production of high quality edible oil is associated with its deterioration in storage, principally as a result of hydrolysis of the oil in the bran.

In 1903 Browne (3) investigated the formation of free fatty acid in rice bran by the action of lipolytic enzymes. He reported that heating the bran inhibited the hydrolytic action to some degree. Further work was reported by West and Cruz (9) in 1933, who concluded that "the formation of free fatty acids in vegetable oils is due to the action of moisture in the presence of enzymes . . . which act as catalysts and accelerate the hydrolysis of the fats. . . . Heating the bran removes the moisture and stops the destructive action of the enzymes. . . ." It is therefore assumed that the presence of enzymes is necessary to bring about this hydrolytic action since at ordinary temperatures moisture alone is not effective in producing rapid hydrolysis of glycerides.

It is apparent that before appreciable progress can be made toward the commercial production of a high quality edible oil from rice bran, some means must be developed to prevent the rapid formation of free fatty acids in the bran after its removal from the rice and prior to extraction of the oil. The present report deals with a) the determination of the factors affecting the formation of free fatty acids in rice bran during storage, and b) possible methods of minimizing or preventing the formation of these acids.

Materials

The rice brans used in these investigations were: a) bran from standard milled, Southwestern-grown,³ long-grained rough rice of different varieties which are referred to hereinafter as "regular" rice bran

and b) bran from Southwestern-grown, long-grained, "Converted" rice. "Converted" is the term applied to the process of forcing the minerals and water-soluble B complex vitamins from the hulls, bran layer, and germ of the rough rice into the endosperm or kernel by means of hot water applied under pressure, followed by application of heat to gelatinize the starch of the endosperm (7). After conversion the rice is milled in the same manner as untreated rice.

Methods

All of the samples of bran were analyzed for oil (Ba-3-38), moisture (Ba-2-38), and free fatty acids (Aa-6-38) by the procedures of the Official Methods of the American Oil Chemists' Society (2) for oil-seeds excepting that the bran did not require grinding and the free fatty acids were determined on approximately 3.0-g. samples of oil. The stored bran was sampled at intervals and analyzed for its content of free fatty acid. The stored brans were mixed either by hand or by shaking the container before removing samples for analysis. The samples were extracted immediately and a record kept of the date and exact time of extraction.

Results

The Effect of Storage at Different Temperatures on the Formation of Free Fatty Acids. A sample of regular rice bran (9.6% oil, 12.6% moisture) was divided into three portions of approximately 40 pounds each. Each portion was stored in a large, closed can and stored at one of the following temperatures: 1. 3°C. (37°F.), 2. 25°C. (77°F.), 3. 31°C. (88°F.). Figure 1 shows the variation in the rate of formation of free fatty acids with temperature. Reference to the curves in this Figure shows that the content of free fatty acids of the bran was initially low but rose rapidly, especially at the higher temperatures (Curves 1

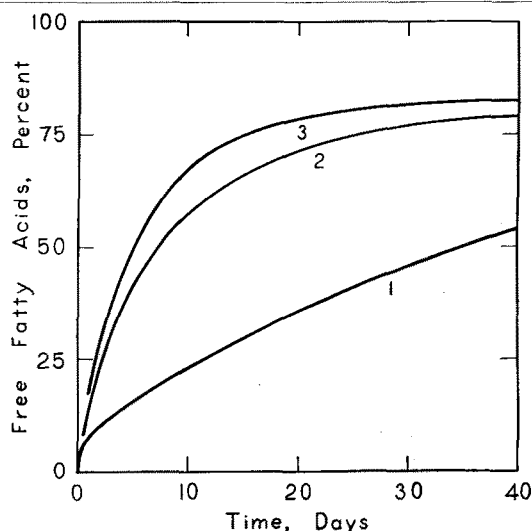


FIG. 1. Effect of storage at different temperatures on the formation of free fatty acids. Curve 1 storage at 3°C., curve 2 at 25°C., and curve 3 at 31°C.

¹ Presented at the 40th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 10-12, 1949; Report of a Study Made Under the Research and Marketing Act of 1946.

² One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

³ Arkansas, Louisiana, and Texas.

and 2, Figure 1). In the initial period of storage, that is the first 12 hours, during which time the rate of formation of free fatty acids is the same in all three samples, thermal equilibrium of the bran with its surroundings is probably not attained. Although all of the samples eventually showed a rapid increase in the content of free fatty acids, the rate of hydrolysis was slowest at the lowest temperature (Curve 1, Figure 1).

Approximately 40 pounds of bran from "Converted" rice (17.27% oil, 9.10% moisture) was stored at a temperature of 3°C. (37°F.) in a 50-pound lard can. This bran stored quite well, increasing from an original content of free fatty acids of 2.6% to 6.5% in 6½ months. The moisture content increased from 9.10% to 9.42% over the same period of time. However, after the transfer of the bran from the lower temperature (3°C.) to a temperature of 25°C., the bran kept well for 7 days, increasing in free fatty acids from 6.5% to 8.1%, after which it deteriorated rapidly, increasing in the content of free fatty acids from 8.1% to 46.9% in 9 days. But bran from freshly milled "Converted" rice stored at 25°C. showed a very gradual increase in free fatty acid content from 3.2% to 10.8% in 6½ months.

Effect of Drying at 70°, 85°, 100°, and 110°C. on the Formation of Free Fatty Acids in Rice Bran During Storage. A sample of freshly milled regular rice bran (15.40% oil, 12.92% moisture, 3.5% initial free fatty acids) was divided into 13 portions. One portion served as a control, and the 12 remaining portions were dried by heating for different periods of time and at different temperatures in a forced draft oven. The times and temperatures used were 1, 2, and 3 hours and 70°C., 85°C., 100°C., and 110°C., respectively. The samples were stored in tightly closed Mason jars at 25°C. Determinations of free fatty acids and moisture were made periodically. The data with respect to the conditions of treatment of the samples and the change in moisture after drying and during storage are given in Table I and curves for the formation of free fatty acids after storage for 122 days in Figure 2. Although a simple mathematical relationship is not evident in the rate curves, comparison of the data in Table I and Figure 2 indicates that the rate of increase in free fatty acids varies directly with the final moisture content of the bran.

Data with reference to the treatment of the samples dried for 1, 2, or 3 hours at 100°C. and 110°C. and the changes in moisture content after drying and during storage are given in Table II and for the rates of formation of free fatty acids during storage for 122 days in Figure 3. Comparison of the data in Table II and Figure 3 indicates that the rates of formation of free fatty acids vary with the final moisture contents of the bran. These results are similar to those obtained by drying the bran at 70°C. and 85°C. The free fatty acid content of the samples

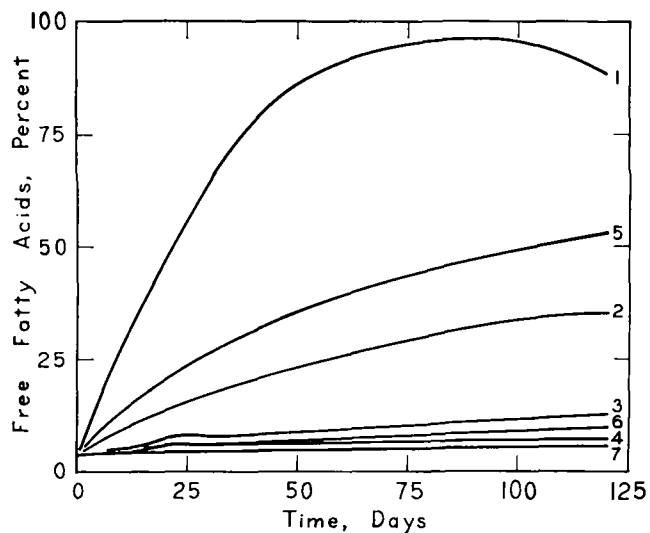


FIG. 2. Effect of drying on the formation of free fatty acids in regular rice bran stored at 25°C. Time and temperatures of drying: (1) None, (2) 1 hr. at 70°, (3) 2 hrs. at 70°, (4) 3 hrs. at 70°, (5) 1 hr. at 85°, (6) 2 hrs. at 85°, (7) 3 hrs. at 85°C.

dried for three hours in each case remained almost stationary throughout the period of storage.

Effect of Drying on the Formation of Free Fatty Acids in Bran From "Converted" Rice. A sample of bran from "Converted" rice (18.01% oil, 6.57% moisture, 3.2% initial free fatty acids) was divided into 13 portions. One portion served as a control, and the

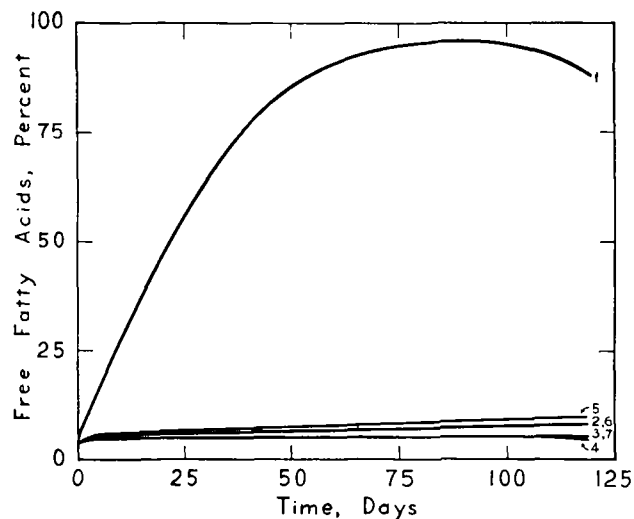


FIG. 3. Effect of drying on the formation of free fatty acids in regular rice bran stored at 25°C. Time and temperatures of drying: (1) None, (2) 1 hr. at 100°, (3) 2 hrs. at 100°, (4) 3 hrs. at 100°, (5) 1 hr. at 110°, (6) 2 hrs. at 110°, (7) 3 hrs. at 110°C.

TABLE I
Regular Rice Bran^a Dried (Heated) at 70°C. and 85°C. and Stored at 25°C.

Experiment No.	1	2	3	4	5	6	7
Treatment							
Heated, hours.....	0	1	2	3	1	2	3
temp., °C.....		70	70	70	85	85	85
Bran temp. after heating, °C.....		57	64	65	64	74	76
Moisture after heating, %.....		7.72	5.11	4.90	10.12	4.80	3.54
Moisture after 18 weeks, %.....	17.40	7.87	5.42	4.16	9.41	4.72	3.50

^a Oil content, 15.40%; original moisture, 12.92%.

TABLE II
Regular Rice Bran^a Dried (Heated) at 100°C. and 110°C. and Stored at 25°C.

Experiment No.	1	2	3	4	5	6	7
Treatment							
Heated, hours.....	0	1	2	3	1	2	3
temp., °C.....		100	100	100	110	110	110
Bran temp. after heating, °C.....		90	94	92	76	104	105
Moisture after heating, %.....		3.63	2.54	2.37	4.41	3.69	2.06
Moisture after 18 weeks, %.....	17.40	4.08	2.44	2.26	3.92	3.66	1.86

^a Oil content, 15.40%; original moisture, 12.92%.

remaining 12 portions were dried for 1, 2, or 3 hours at one of the following temperatures: 1. 70°C., 2. 85°C., 3. 100°C., and 4. 110°C. The bran was dried in trays in a forced draft oven and stored in tightly closed Mason jars at 25°C. The contents of free fatty acids and moisture were determined periodically.

The data with reference to variation in the content of free fatty acids as a function of the moisture con-

TABLE III
Storage of Dried (Heated) "Converted" Rice Bran^a at 25°C. for 130 and 200 Days

Heat treatment	Moisture, %	Moisture after 7 months, %	F. F. A. after 130 days, %	F. F. A. after 200 days, %
None.....	6.57	6.80	8.8	10.8
70°C.				
1 hr.....	3.52	6.02	3.6	7.9
2 hrs.....	3.05	4.41	3.7	4.5
3 hrs.....	2.97	3.91	3.3	3.6
85°C.				
1 hr.....	3.10	5.06	3.7	7.7
2 hrs.....	2.02	3.42	3.3	4.7
3 hrs.....	2.16	2.97	3.3	4.7
100°C.				
1 hr.....	2.62	4.04	3.6	9.2
2 hrs.....	1.65	2.58	3.4	4.8
3 hrs.....	1.50	3.21	3.3	7.5
110°C.				
1 hr.....	4.00	5.18	3.9	6.9
2 hrs.....	1.82	2.95	3.6	5.8
3 hrs.....	1.29	2.01	2.8	4.4

^a Oil content, 18.01%, original F. F. A., 3.2%.

tent of the bran after storage for 130 and 200 days are given in Table III. Reference to this table indicates that the moisture contents increased during storage and that in each case the less hydrated samples increased in fatty acid content at the slower rate. Since the rate of formation of free fatty acids was only slightly affected by drying over the temperature range investigated, data for only three experiments are reproduced in the form of curves, namely, 1. the bran dried for 3 hours at 70°C., 2. the bran dried 1 hour at 70°C., and 3. the control (Figure 4).

Effect of Storage at Different Relative Humidities on the Formation of Free Fatty Acids in Rice Bran. Six samples of bran, three from freshly milled regular rice and three from "Converted" rice, were stored in large desiccators containing saturated salt solutions to maintain relative humidities of 32.5%, 53.3%, and 75.4% (5). The bran contained in large crystallizing dishes was placed in the desiccators and stored at 25°C. The effect of the relative humidity on the moisture content of the stored bran is shown in Table IV, and the effect on the rate of formation of free fatty acids in the bran from "Converted" rice in Figure 5. From Figure 5 it is evident that equilibrium was attained within five days. The rate of hydrolysis increased proportionally to the relative humidity of storage and therefore to the final moisture content. The results agree with those obtained on bran from regular rice (Figure 6). In the case of regular rice

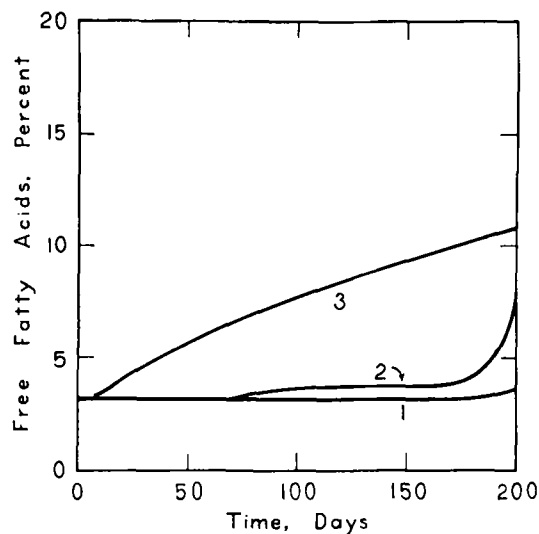


FIG. 4. Effect of drying on the formation of free fatty acids in bran from "Converted" rice stored at 25°C. Time and temperature of drying: (1) 3 hrs. at 70°, (2) 1 hr. at 70°C., (3) None.

bran equilibrium was also attained within five days. Although inhibition of hydrolysis in the regular bran was not as great as that from "Converted" rice, the results were similar.

Effect of Storage at Different Relative Humidities on the Formation of Free Fatty Acids in Predried Bran From Regular and "Converted" Rice. Since both drying and the relative humidity of storage markedly affected the formation of free fatty acids

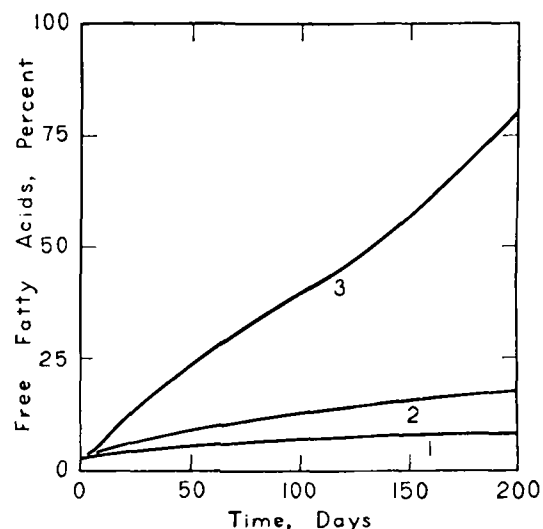


FIG. 5. Effect of storage at different relative humidities on the formation of free fatty acids in bran from "Converted" rice stored at 25°C. Relative humidity: (1) 32.5%, (2) 53.3%, and (3) 75.4%.

TABLE IV
Storage of Rice Bran at 25°C. and Different
Relative Humidities

Experiment No.	1	2	3	4	5	6
Type of bran.....	"Converted"			Regular		
Oil content, %.....	17.27	17.27	17.27	14.90	14.90	14.90
Original moisture, %.....	9.10	9.10	9.10	11.28	11.28	11.28
Relative humidity, %.....	32.5	53.3	75.4	32.5	53.3	75.4
Moisture after storage, % ^a	6.15	7.25	8.60	8.44	9.88	11.18

^a "Converted" stored 11 weeks; regular stored 6 weeks.

in rice bran, an examination was made of the rate of hydrolysis of dried samples of the bran from regular and "Converted" rice stored at different relative humidities. A sample of regular rice bran (15.40% oil, 12.92% moisture, 3.5% initial free fatty acids) was divided into 9 portions. One portion served as a control; four portions were dried in a tray in a forced draft oven for 3 hours at 85°C.; and the remaining four samples were dried in a forced draft oven for 2 hours at 110°C. Of the samples dried for 3 hours at 85°C., one served as a predried control and the others were stored at various relative humidities. The samples dried for 2 hours at 110°C. were similarly treated, that is, one served as predried control for comparison with the others which were stored at specific relative humidities. As can be seen from the data in Table V, the moisture content of the samples increased during storage and from Figure 7 that the increase in free fatty acids varied with the relative humidity during storage and also with the final moisture content of the bran.

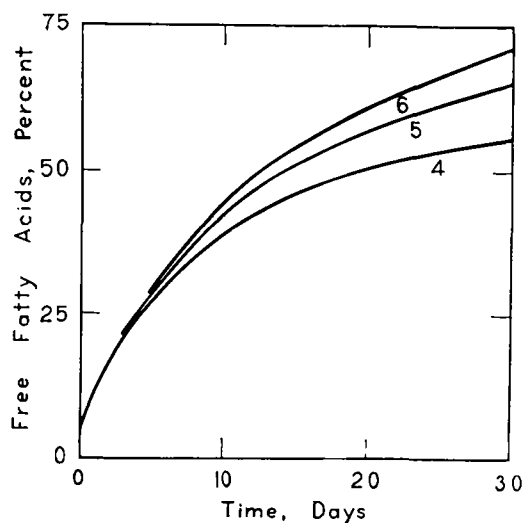


FIG. 6. Effect of storage at different relative humidities on the formation of free fatty acids in regular rice bran stored at 25°C. Relative humidity: (4) 32.5%, (5) 53.3%, and (6) 75.4%.

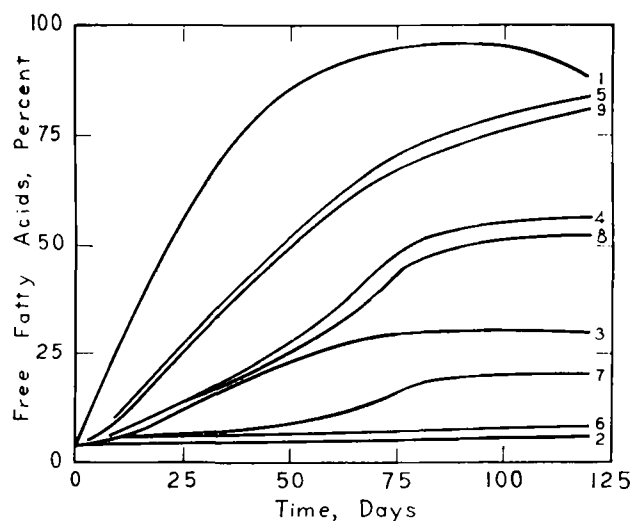


FIG. 7. Effect of storage at various controlled relative humidities on the formation of free fatty acids in predried regular rice bran stored at 25°C. Time and temperature (°C.) of drying and relative humidity of storage: (1) 0, 0, uncontrolled, (2) 3 hrs., 85°, uncontrolled, (3) 3 hrs., 85°, 32.5%, (4) 3 hrs., 85°, 53.3%, (5) 3 hrs., 85°, 75.4%, (6) 2 hrs., 110°, uncontrolled, (7) 2 hrs., 110°, 32.5%, (8) 2 hrs., 110°, 53.3%, (9) 2 hrs., 110°, 75.4%.

A similar experiment was made with bran from "Converted" rice (18.01% oil, 6.57% moisture, 3.2% initial free fatty acids). The times and temperatures employed for drying were 5 hours at 85°C. and 2 hours at 110°C. As can be seen from Table VI, the moisture contents increased during storage, but all of the samples kept well, those having the lowest moisture content keeping best.

Effect of Added Moisture on the Formation of Free Fatty Acids in Brans From Regular and "Converted" Rice. A sample of bran from "Converted" rice (18.01% oil, 6.57% moisture, 3.2% initial free fatty acids) was divided into six portions. One portion served as a control, and four of the remaining five portions were dried in a tray in a forced draft oven for 2 hours at 85°C. All of the samples were stored in tightly closed Mason jars at 25°C. Representative data obtained in this experiment are presented in Figure 8.

Curve 1 of Figure 8 represents the undried control. To the second undried sample, represented by Curve 2, enough moisture was added to increase the moisture content of the bran to 1.5 times that of the original. In this sample the content of free fatty acids increased sharply and then gradually decreased. In the predried sample represented by Curve 3 the content of free fatty acids remained stationary throughout the storage period. The sample of dried bran which was stored for 120 days and then restored to

TABLE V
Dried (Heated) Regular Rice Bran^a Stored at 25°C. and Different Relative Humidities

Experiment No.	1	2	3	4	5	6	7	8	9
Treatment									
Heated, hours.....	0	3	3	3	3	2	2	2	2
temp., °C.....		85	85	85	85	110	110	110	110
Bran temp. after heating, °C.....		76	76	76	76	104	104	104	104
Moisture after heating, %.....		3.54	3.54	3.54	3.54	3.69	3.69	3.69	3.69
Relative humidity, %.....			32.5	53.3	75.4		32.5	53.3	75.4
Moisture after 1 week, %.....	13.45		5.10	6.54	7.72		3.44	5.34	7.33
Moisture after 9 weeks, %.....	14.76		6.52	8.81	12.56		5.42	8.05	12.13
Moisture after 18 weeks, %.....	17.40	3.50	6.76	8.96	13.18	3.66	6.10	8.44	12.70

^a Oil content, 15.40%; original moisture, 12.92%.

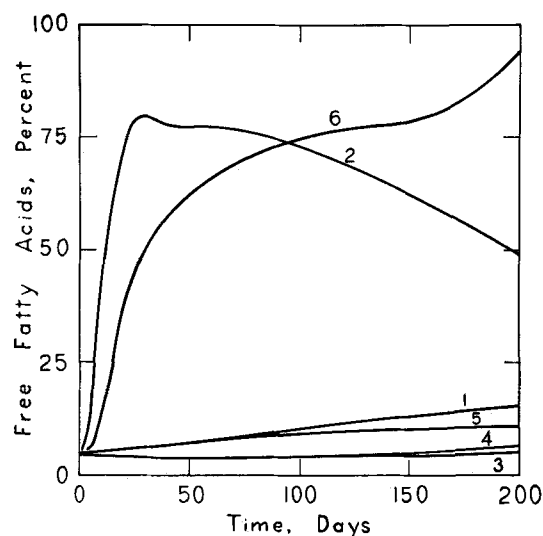


FIG. 8. Effect on the formation of free fatty acids in bran from "Converted" rice predried for 2 hrs. at 85°C., conditioned by addition of moisture, and stored at 25°C., (1) Original sample, (2) original raised to 1½ times original moisture, (3) predried, no added moisture, (4) predried, stored 120 days, restored to original moisture, (5) predried, restored to original moisture, (6) predried, raised to 1½ times original moisture.

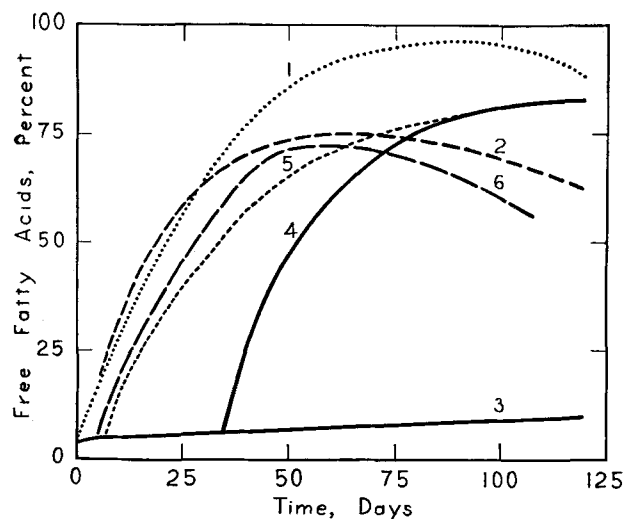


FIG. 9. Regular rice bran treated exactly as described in Fig. 8, except (4) was stored 35 days and then restored to original moisture.

its original moisture content is represented by Curve 4 which shows that an increase in hydrolysis followed the addition of moisture. To Sample 5, represented by Curve 5, enough moisture was added immediately after drying to restore it to its original moisture content. In this case the rate of hydrolysis is similar to that of the control, Curve 1. In Sample 6 enough moisture was added to increase its moisture content to 1.5 times that of the original. The curve for this sample, like that (Curve 2) of the undried sample which also had its moisture content increased to 1.5 times the original, shows a rapid increase in the rate of hydrolysis compared with the undried control.

This experiment was repeated with regular rice bran, as shown by the data in Figure 9 in which the designation of the curves correspond to those given previously for the bran from "Converted" rice. The two experiments were again repeated with bran from both "Converted" and regular rice, employing the time and temperature of 2 hours at 110°C., with the results shown in Figures 10 and 11, respectively.

Effect of Chemical Treatment and of Inert Atmosphere on the Formation of Free Fatty Acids in Stored Rice Bran. Five samples of 5.5 pounds each of bran from freshly milled regular rice (16.8% oil) were

stored in stainless steel buckets, one control sample and four experimental samples, each containing a different inhibitor (1). Each of the samples was treated with one of the following inhibitors: 0.31% of ethylene chlorohydrin, 0.03% of sodium cyanide, 0.19% of propylene glycol dipropionate, or 0.03% of 1,3-dimethyl-4,6-bis(chloromethyl) benzene. After storage for seven days at 25°C., all of the samples had deteriorated. The control sample had increased in free fatty acid content from 3.3% to 77.1%, and the bran containing the inhibitor which gave the best results, that is, sodium cyanide, increased in free fatty acids from 3.3% to 68.0%.

Four samples of freshly milled regular rice bran (16.66% oil, 11.62% moisture) were treated as follows:

- A portion of the bran was placed in a large crystallizing dish, which was in turn placed in a vacuum desiccator and stored under vacuum at room temperature.
- A second portion of the bran was placed in a two-quart Mason jar. Carbon dioxide was passed into the head space, and the jar was closed and stored at room temperature.
- A five-liter flask was half-filled with a third portion of the bran, and nitrogen gas, purified by passing it over hot copper, was passed through the bran after which the flask was closed and stored at room temperature.
- A fourth and control portion of bran was stored at room temperature in a 50-pound lard can.

After storage for 23 days all four samples were found to have a content of free fatty acids of 39%

TABLE VI
Dried (Heated) "Converted" Rice Bran^a Stored at 25°C. and Different Relative Humidities

Experiment No.	1	2	3	4	5	6	7	8	9
Treatment, Heated, hours.....	0	5	5	5	5	2	2	2	2
temp., °C.....	85	85	85	85	85	110	110	110	110
Bran temp. after heating, °C.....	81	81	81	81	81	88	88	88	88
Moisture after heating, %.....	2.05	2.05	2.05	2.05	2.05	1.82	1.82	1.82	1.82
Relative humidity, %.....	32.5	53.3	75.4	32.5	53.3	75.4
Moisture after 4 weeks' storage, %.....	3.78	4.47	5.16	4.14	5.20	5.36
Moisture after 16 weeks' storage, %.....	4.36	5.38	6.77	4.30	5.33	6.70
Moisture after 28 weeks' storage, %.....	6.80	2.94	4.99	6.13	7.16	2.95	5.04	5.62	7.14
Free Fatty Acid, %									
Days storage 0.....	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
10.....	3.5	3.2	3.3	3.4	3.4	3.0	3.4	3.4	3.4
60.....	6.0	3.2	3.6	4.0	4.5	3.0	3.7	4.2	4.3
100.....	7.7	3.3	4.0	4.5	5.2	3.5	4.0	4.9	5.2
150.....	9.2	3.2	4.0	4.9	5.3	6.6

^a Oil content, 18.01%; original moisture, 6.57%.

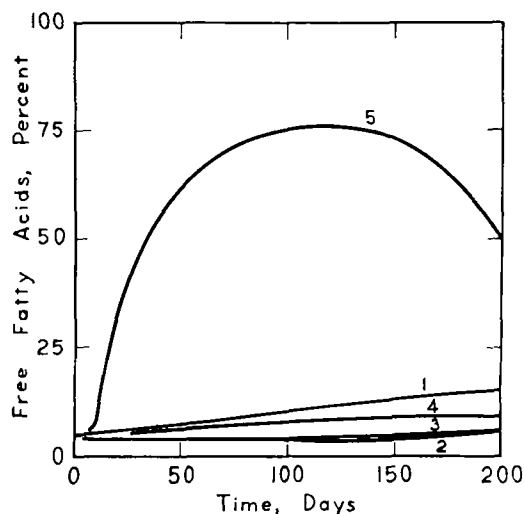


FIG. 10. Effect on the formation of free fatty acids in bran from "Converted" rice predried for 2 hrs. at 110°C., conditioned by addition of moisture, and stored at 25°C., (1) Original sample, (2) predried, no added moisture, (3) predried, stored 120 days, restored to original moisture, (4) predried, restored to original moisture, (5) predried, raised to 1½ times original moisture.

to 42%. After 50 additional days of storage all four samples had contents of free fatty acids of 64% to 78%. The best sample (free fatty acids content 64%) was stored under vacuum and therefore was partially dehydrated during the storage period. The moisture content of this sample decreased from 11.44% to 10.22% whereas the moisture content of the other samples remained unchanged.

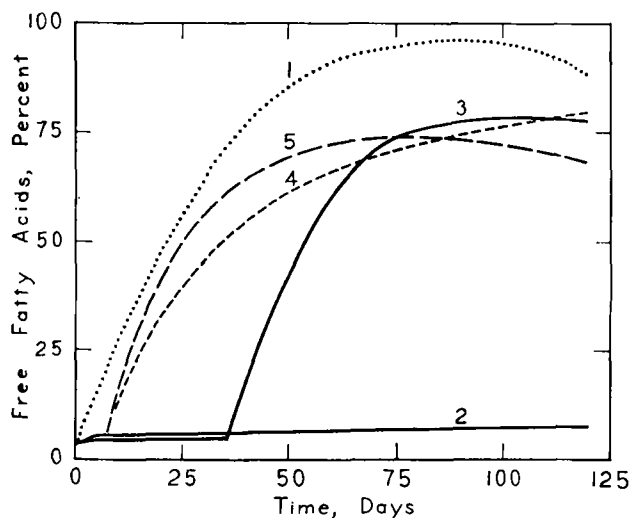


FIG. 11. Regular rice bran treated exactly as described in Fig. 10. except (3) was stored 35 days and then restored to original moisture.

Summary and Conclusions

The oil, contained in bran from regularly milled rice, when stored at prevailing atmospheric temperature, humidity, and natural moisture content is subject to rapid hydrolysis which increases the free fatty acid content of the oil to a point where it cannot be economically refined.

Data have been presented showing the effects of a) temperature, b) drying at temperatures of 70°, 85°, 100°, and 110°C. for various periods of time up to 5 hours, c) different relative humidities before and after drying, and d) added moisture on the rate of formation of free fatty acids during storage in bran from both regular and "Converted" rice.

Decreasing the storage temperature tends to retard the formation of free fatty acids. In the case of regular rice bran deterioration during storage occurred at a fairly rapid rate even at 3°C. whereas bran from "Converted" rice was fairly stable when stored at this temperature.

The investigation of the effect of heating or drying and the effect of different relative humidities on the storage of rice bran have shown that bran from both regular and "Converted" rice can be stored for periods of at least four months without excessive increase in the content of free fatty acids, provided the bran is dried sufficiently and is maintained at a low moisture content. An increase in the moisture content of predried bran causes a rapid increase in the free fatty acid content of the oil in the bran.

Investigations of the effect of chemical inhibitors and of inert atmosphere on the rate of free fatty acid formation of regular rice bran indicated that these were ineffective in preventing deterioration.

Acknowledgment

The authors wish to acknowledge the technical assistance of M. E. Curet and M. C. Curet in carrying out some of the analyses reported here, of Converted Rice inc., Houston, Texas, for furnishing samples of bran from "Converted" rice, and of United Rice Milling Products Company inc., New Orleans, Louisiana, for making available supplies of freshly milled regular rice bran.

REFERENCES

1. Altschul, A. M., *The Cotton Gin and Oil Mill Press*, 50, No. 1, A-2-A-10 (1949).
2. American Oil Chemists' Society. *Official and Tentative Methods*, 2nd ed., ed. by V. C. Mehlenbacher, Chicago, 1946.
3. Browne, Jr., C. A., *J. Am. Chem. Soc.*, 25, 948-954 (1903).
4. Feuge, R. O., and Reddi, P. B. V., *J. Am. Oil Chem. Soc.*, 26, 349-353 (1949).
5. Karon, M. L., and Adams, M. E., *J. Am. Oil Chem. Soc.*, 25, 21-22 (1948).
6. Kik, M. C., and Williams, R. R., *Bull. Nat. Res. Council*, No. 112, 30-36, June, 1945.
7. O'Donnell, W. W., *Food Industries*, 19, 763-768, 894, 896 (1947).
8. Reddi, P. B. V., Murti, K. S., and Feuge, R. O., *J. Am. Oil Chem. Soc.*, 6, 206-211 (1948).
9. West, A. P., and Cruz, A. O., *Philippine J. Sci.*, 52, 1-78 (1933).