

# Changes in Pearl Millet Meal During Storage<sup>1</sup>

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## ABSTRACT

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Pearl millet meal was stored at 19°C, 58% rh; 27°C, 64% rh; or 42°C, 75% rh. Changes in sensory attributes (odor), mold count, fatty acid composition, total titratable acidity, and peroxide value were followed. A trained panel detected changes in the odor of stored millet after 108, 60, and 12 hr for storage temperatures 19, 27, and 42°C, respectively. A reduction in mold count during the first six weeks of storage suggested that fungal growth had little effect on odor changes observed in the first week of storage.

However, the times at which odor changes were detected in all stored samples corresponded to a fat acidity level of 30 mg of KOH per 100 g of meal and coincided with the end of the induction period for peroxide formation. Thin-layer and gas chromatographic studies further confirmed that alterations in lipid components were responsible for rapid quality deterioration in pearl millet meal.

A number of traditional and mechanized processes are used to mill pearl millet (Goussault and Adrian 1977, Rasper 1977, Wyss 1977), with extraction rates from about 75% for the SOTRAMIL process (Goussault and Adrian 1977) to about 85% for wooden-mortar milling. None of the milling processes completely separates the germ from the rest of the kernel; the lipids in the germ can therefore be adsorbed by the components of the milled products, which lowers the flour's keeping quality. Based on chemical analyses, Carnovale and Quaglia (1973) suggested that the rapid deterioration in quality of pearl millet flour (16.3% moisture) during storage for three months at 30°C stems mainly from hydrolytic rather than oxidative decomposition of lipids. Thiam et al (1976) stored millet flours for 17 days at 30°C with 50 and 75% relative humidities and found that microbial growth did not occur, nor did off-flavors or odors. In addition, lipid oxidation was not detected by those authors. Details of the sensory evaluation of stored flours were not reported by Carnovale and Quaglia (1973) or Thiam et al (1976), so the relationship between chemical and organoleptic changes could not be assessed. We describe organoleptic evaluations of stored pearl millet meal that indicate both hydrolytic and oxidative changes in millet lipid components during the first week of storage.

## MATERIALS AND METHODS

### Materials

We used an HMP550 (Tift 23DB<sub>1</sub>/\*PII85642) random mating bulk pearl millet population from the Branch Experiment Station, Hays, KS. The whole grain was ground in an experimental roller mill to pass a 50 GG screen. The meal was stored in replicates in cotton bags under the following storage condition: 19°C, 58% rh; 27°C, 64% rh; and 42°C, 75% rh. After designated storage periods, samples were taken from the bags by a hollow tube probe.

### Methods

**Moisture Content and Mold Count.** Standard AACC methods (1962) were used for the moisture determinations and mold counts.

**Sniff Test.** The sniff test was conducted with seven selected panelists (three females, four males) with previous sensory evaluation experience. They were first acquainted with the aroma characteristics of the fresh and aged millet meal samples, as a frame of reference.

Samples taken at the same times (0, 12, 36, 60, 84, and 108 hr) from the three storage conditions were presented to the panel members in odor-free glass jars. Six coded samples were presented

in a random order at each sitting. Panelists were asked to sniff the samples one at a time and compare them with the reference samples. Results and comments were recorded on a score sheet consisting of the words "fresh" and "aged" separated by a horizontal line. The panelists were asked to mark an "X" at the point on the line that best described their feelings about the sample. Each sniff test value was derived by measuring the distance in centimeters from the "fresh" reference point to the point marked by the panelist.

**Thin-Layer and Gas Chromatography.** Free and bound lipids were extracted, fatty acids esterified, and methyl esters determined by gas chromatography. Free lipids from samples stored 0, 60, and 108 hr were also characterized by thin-layer chromatography. Methods for lipid analysis were previously described (Lai and Varriano-Marston 1980).

**Fat Acidity and Peroxide Value.** Fat acidity was determined by the AOAC rapid method (1960). For peroxide value determinations, 5-g samples were mixed with 50 ml of chloroform in a Stein mill for 3 min. The peroxide value of the filtrate was determined by the method of Takagi et al (1978). Replicate samples for fat acidity and peroxide value determinations were taken after the same time periods as were the odor evaluations.

**Histology.** Millet kernels were soaked in distilled water, and lipase location was determined by the Tween (Bancroft 1975) and  $\beta$ -naphthylaurate (Sastrey et al 1977) methods.

**Statistical Analyses.** An analysis of variance was run on the data (Snedecor and Cochran 1967), and Duncan's (1955) multiple range test was applied to determine significant differences among means.

## RESULTS AND DISCUSSION

### Mycology

Microflora affect the quality of cereal grains during storage. Fungal growth is often related to increases in fat acidity (Baker et al 1959), and fungal lipases are responsible for the increases in free fatty acids (Dirks et al 1955, Goodman and Christensen 1952), so fungal growth must be determined in all storage studies involving grains.

TABLE I  
Mold Counts<sup>a</sup> in Stored Pearl Millet Meal

Time (weeks)	Storage Condition		
	19°C, 58% rh	27°C, 64% rh	42°C, 75% rh
Fresh	14,800	14,800	14,800
3	10,800	11,250	400
6	10,200	6,100	200
9	10,750	4,100	1,250
12	5,150	3,000	31,800

<sup>a</sup>Number of mold colonies per gram of sample.

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In microbiological studies of our stored millet meals, the samples contained normal levels of field fungi typically found in cereal grains grown in Kansas. *Alternaria*, *Fusarium*, and *Cladosporium* were the major fungi but the genera *Mucor*, *Phoma*, *Aspergillus*, and *Penicillium* also were identified in small numbers in some samples.

For all storage conditions, mold counts declined during the first six weeks of storage (Table I). Therefore, fungal growth probably had little effect on quality deterioration in millet meal during the early weeks of storage.

After nine weeks, only samples stored at 42°C, 75% rh showed

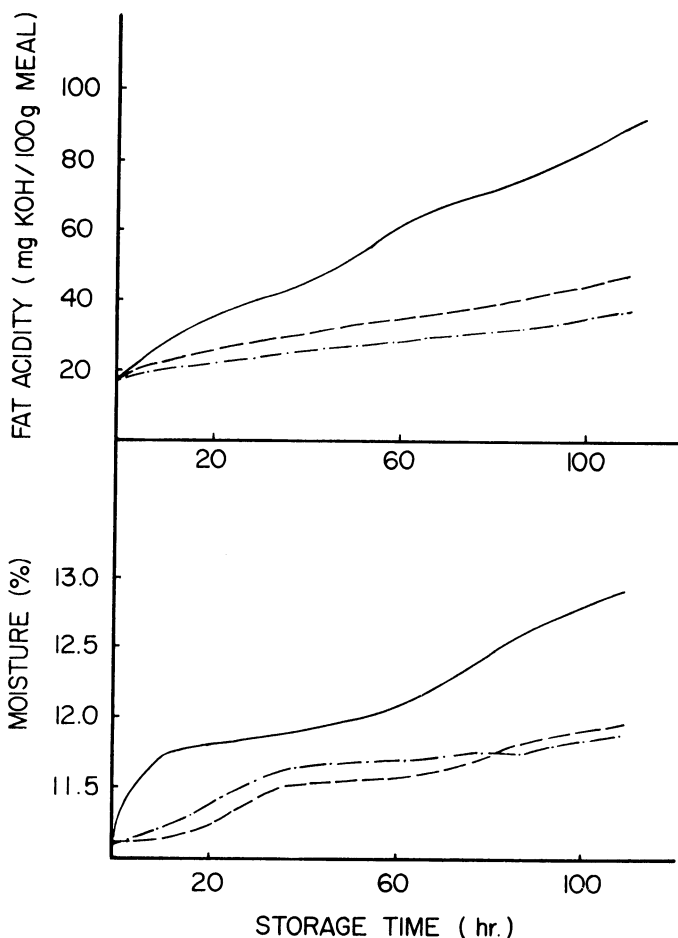


Fig. 1. Changes in fat acidities and moisture contents of millet meal during storage. — = 42°C, 75% rh; --- = 27°C, 64% rh; - · - · - = 19°C, 58% rh.

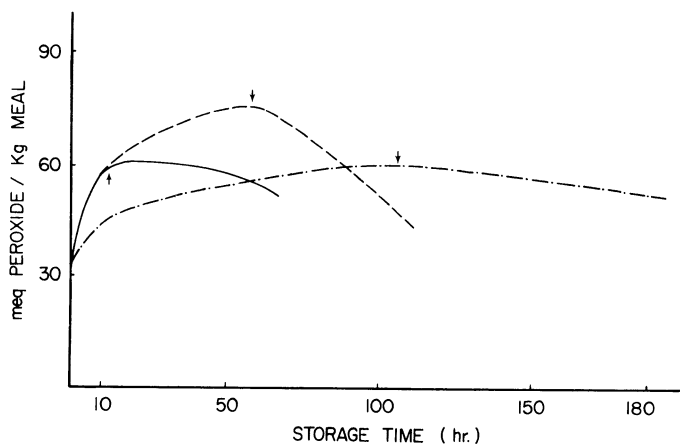


Fig. 2. Changes in peroxide values of millet meal during storage. Arrows designate time when panelists first detected an odor change. — = 42°C, 75% rh; --- = 27°C, 64% rh; - · - · - = 19°C, 58% rh.

increased mold counts, with *Penicillium* the predominant species (Table I). Storage fungi like *Penicillium* grow at equilibrium relative humidities of 70–90%. Conversely, field fungi require high moisture conditions for growth and die off rapidly in grains held with moisture contents in equilibrium with relative humidities of 70–75% (Lutey and Christensen 1963).

### Sniff Test

Reports from India<sup>3</sup> suggested that millet could be kept for only a few days after grinding, so we had our panelists do odor evaluations to see if quality deterioration could be detected during the first five days of storage.

During the taste panel orientation, freshly ground millet and millet meal that had been stored at 42°C for more than three months were studied by the panelists. They described the aroma of the Fresh sample as fresh, green, and sweet, resembling creamed corn; the Aged sample was described as sour or acidic, musty, and dry and dusty.

Marked disappearance of sweet aromas paralleled the increased appearance of dusty and oxidized characteristics in the stored samples. In addition, the panel noticed that high temperatures and humidities shortened the time necessary before “Aged” characters were detected. The olfactory data showed significant ( $P < 0.01$ ) changes in the odor response of panelists for samples stored 12 hr at 42°C, 75% rh. On the other hand, 60 and 108 hr, respectively, were required before significant odor changes were noted for millet meals stored at 27°C, 64% rh and 19°C, 58% rh.

<sup>3</sup>Reddy, Geeravani. 1978. Home Science College, Hyderabad, India. Personal communication.

TABLE II  
Changes in Fatty Acid Composition (%) of Free Lipids of Pearl Millet During Storage at 42°C, 75% rh<sup>a</sup>

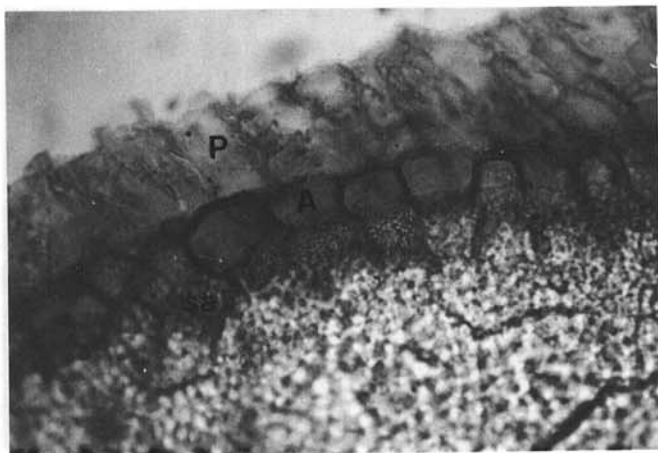
Fatty Acid	Storage Time, hr		
	Fresh	60	108
C <sub>14:0</sub>	trace	0.09	0.08
C <sub>16:0</sub>	21.56 a	20.83 a	19.19 b
C <sub>16:1</sub>	0.96 a	1.19 a	1.19 a
C <sub>18:0</sub>	7.32 a	7.38 a	7.82 a
C <sub>18:1</sub>	28.20 a	28.10 a	29.85 b
C <sub>18:2</sub>	38.78 a	38.21 a	37.53 a
C <sub>18:3</sub>	2.36 a	3.45 ab	3.75 b
C <sub>20:0</sub>	1.04 a	0.78 b	0.61 b
C <sub>22:0</sub>	trace	trace	trace

<sup>a</sup> Within each fatty acid, values with different letters differ significantly ( $P < 0.05$ ).

TABLE III  
Changes in Fatty Acid Composition (%) of Bound Lipids of Pearl Millet During Storage at 42°C, 75% rh<sup>a</sup>

Fatty Acid	Storage Time, hr		
	Fresh	60	108
C <sub>10:0</sub>	1.18 a	7.44 b	6.41 b
Unknown	0.85 a	0.34 a	0.68 a
C <sub>12:0</sub>	1.05 a	4.28 b	7.08 b
C <sub>13:0</sub>	0.62 a	0.65 a	2.20 b
C <sub>14:0</sub>	0.42 a	0.18 a	0.30 a
C <sub>15:0</sub>	0.73 a	2.60 b	2.72 b
C <sub>16:0</sub>	21.11 a	21.65 a	21.19 a
C <sub>16:1</sub>	0.96 a	0.59 b	0.51 b
C <sub>17:0</sub>	0.23 a	0.52 b	0.90 c
C <sub>18:0</sub>	5.12 a	3.27 b	3.23 b
C <sub>18:1</sub>	19.49 a	17.65 a	15.22 b
C <sub>18:2</sub>	29.37 a	28.08 a	26.52 b
C <sub>18:3</sub>	3.16 a	2.13 a	1.32 b
C <sub>20:0</sub>	7.81 a	1.19 b	0.89 b
C <sub>24:0</sub>	7.87 a	9.38 b	10.67 b

<sup>a</sup> Within each fatty acid, values with different letters differ significantly ( $P < 0.05$ ).



**Fig. 3.** Histochemical location of lipase activities in the pericarp (P), aleurone (A), and subaleurone (SA) layers of pearl millet.

### Thin-Layer and Gas Chromatography

Sensory evaluation data indicated that millet meal quality changed early in storage. We wanted to determine if alterations in lipid components were related to those changes.

Thin-layer chromatography of free nonpolar lipids extracted from millet meal stored for up to 108 hr showed progressive increases in free fatty acid and partial glyceride components during storage, indicating hydrolytic breakdown of triacylglycerols.

Changes in the fatty acid composition of pearl millet's free and bound lipids during storage at 42°C, 75% rh were determined and are presented in Tables II and III, respectively. Although fatty acid composition did not change qualitatively during storage, quantities of fatty acids changed in both free and bound lipid fractions, more in bound than in free lipids. For example, of the C<sub>18</sub> acids, oleic and linolenic acids of the free lipid fraction increased slightly after 108 hr of storage (Table II), whereas all bound C<sub>18</sub> acids decreased during storage (Table III). Reductions in unsaturated acids of the bound lipids are indicative of oxidative changes.

### Fat Acidity

Hydrolytic changes in lipids during storage were also followed by determining fat acidity values. Fat acidity and moisture content of millet meal stored at 42°C, 75% rh increased more rapidly during the first 108 hr of storage than did those of samples stored at 27°C, 63% rh or 19°C, 58% rh (Fig. 1). The time at which odor changes were detected in samples from all storage conditions corresponded to a fat acidity level of 30 mg of KOH per 100 g of meal. The high moisture content of samples stored at 42°C may have accelerated development of both fat acidity and objectionable odors.

### Peroxide Value

Peroxide values, which indicate oxidative changes in lipids, steadily increased early in storage, reached a maximum, and then gradually declined under all three storage conditions (Fig. 2). However, the rates at which peroxide values changed differed for each storage treatment. Samples stored at 42°C, 75% rh terminated the induction period earlier than those stored at either 27°C, 64% rh or 19°C, 58% rh. For each storage condition, the end of the induction period coincided with the time when panelists first detected an odor change.

### Histology

The data show that hydrolytic and oxidative changes in millet lipid components parallel odor changes observed by panelists. One

way to reduce lipid changes during storage would be to remove the principal parts of the kernels that contain lipids: the pericarp, aleurone, and germ (Lai and Varriano-Marston 1980). Decortication studies by De Francisco et al<sup>4</sup> indicate that pearl millet pericarp is easily removed but that germ portions remain attached to the kernels. Our histochemical studies on pearl millet kernels indicated that lipase activity was located mainly in the germ, pericarp, and aleurone and subaleurone layers (Fig. 3). Decortication procedures would probably reduce lipid changes during storage, but the germ remaining in decorticated kernels would assure continued oxidative and lipolytic activity.

<sup>4</sup>De Francisco, A., Shepherd, A., Hoseney, R. C., and Varriano-Marston, E. 1980. Unpublished.

### ACKNOWLEDGMENTS

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### LITERATURE CITED

- AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1962. Approved Methods of the AACC (7th ed.) Am. Assoc. Cereal Chem.: St. Paul, MN.
- ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. 1960. Official Methods of Analysis (9th ed.) Assoc. Off. Anal. Chem.: Washington, DC.
- BAKER, D., NEUSTADT, M. H., and ZELENY, L. 1959. Relationships between fat acidity values and types of damage in grain. *Cereal Chem.* 36:308.
- BANCROFT, J. D. 1975. *Histochemical Technique* (2nd ed.) Butterworth & Co. Ltd.: London.
- CARNOVALE, E., and QUAGLIA, G. B. 1973. Influence of temperature and humidity controlled storage on the chemical composition of milling products from millet. *Ann. Technol. Agric.* 22:371.
- DIRKS, B. M., BOYER, P. D., and GEDDES, W. F. 1955. Some properties of fungal lipases and their significance in stored grain. *Cereal Chem.* 32:356.
- DUNCAN, D. B. 1955. New multiple range and multiple F tests. *Biometrics.* 11:1.
- GOODMAN, J. J., and CHRISTENSEN, C. M. 1952. Grain storage studies. XI. Lipolytic activity of fungi isolated from stored corn. *Cereal Chem.* 29:299.
- GOUSSAULT, B., and ADRIAN, J. 1977. The milling of Pennisetum millet and the value of the protein in the products. Page 13 in: Dendy, D. A. V., ed. *Proceedings of a Symposium on Sorghum and Millet for Human Food.* Tropical Products Inst.: London.
- LAI, C. C., and VARRIANO-MARSTON, E. 1980. Lipid content and fatty acid composition of free and bound lipids in pearl millets. *Cereal Chem.* 57:271.
- LUTEY, R. W., and CHRISTENSEN, C. M. 1963. Influence of moisture content, temperature, and length of storage upon survival of fungi in barley kernels. *Phytopathology* 53:713.
- RASPER, V. F. 1977. Palyi's compact system for debranning of sorghum and millet. Page 59 in: Dendy, D. A. V., ed. *Proceedings of a Symposium on Sorghum and Millet for Human Food.* Tropical Products Inst.: London.
- SASTRY, B. S., RAMAKRISHNA, M., and RAGHAVENDRA, M. R. 1977. Histochemical localization of lipase in the rice grain. *J. Food Sci. Technol.* 14:273.
- SNEDECOR, G. W., and COCHRAN, W. G. 1967. *Statistical Methods* (6th ed.) Iowa State University Press: Ames.
- TAKAGI, T., MITSUNO, Y., and MASUMURA, M. 1978. Determination of peroxide value by the colorimetric iodine method with protection of iodide as cadmium complex. *Lipids* 13:147.
- THIAM, A. A., DRAPRON, R., and RICHARD-MOLARD, D. 1976. Causes d'alteration des farines de millet et de sorgh. *Ann. Technol. Agric.* 25(3):253.
- WYSS, E. 1977. Millet and sorghum milling. Page 111 in: Dendy, D. A. V., ed. *Proceedings of a Symposium on Sorghum and Millet for Human Food.* Tropical Products Inst.: London.