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The Effect of Variations in Moisture Content on the Storage Deterioration Rate of Cake Mixes[a]

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The effect of moisture content on the storage life of chemically leavened cake mixes (white, yellow, gingerbread, devil's food) is studied. Physical properties of cakes prepared from stored mixes are shown to undergo change as a result of premature reaction of leavening ingredients. An increase in free fatty acids is noted in stored mixes of moderate to high moisture contents. Products formulated with flour of 7.4% moisture content or less were generally resistant to deleterious changes during storage.

The great increase in popularity of prepared cake mixes within the last decade has made the storage stability of these products a matter of prime importance to a large segment of the cereal industry. The first mixes placed on the market had a very short shelf life and frequently became unusable within a few weeks or months after manufacture. It is known that one of the chief factors affecting the stability of these mixes is a loss of leavening power due to a premature reaction of the sodium bicarbonate with the acidic components of the mix *(6)*. The rate of this reaction is greatly affected by the moisture content of the other mix ingredients, especially the flour *(7)*. Flour moisture is also known to affect the rate at which free fatty acids develop, and relatively large quantities of free fatty acids are believed to affect the baking quality of flour *(3)*.

Considerable work has been done on the relationship between moisture content of flour and the rate at which its suitability as a bread ingredient decreases during storage. Cuendet *et al., (2)* found that the stability of various grades of wheat flour stored in sealed containers at 37.8° C. decreased with decreasing refinement and with increasing moisture content. Greer, Jones, and Moran *(4)* found that flour of 13-14% moisture content stored up to 10 years at ordinary temperatures retained satisfactory baking quality when the ash content was below 0.6%, the protein content was below 11%, and the flour was packed in non-gas-tight containers. The conclusions reached by these workers with bread flour are no doubt applicable, in some degree, to cake mixes, but there are special problems connected with the latter product which have heretofore remained unexplored.

These considerations suggested that a definitive study of the effect of moisture content on the storage life of cake mixes would be of value in determining specification requirements. To this end, four varieties of cake mix, each utilizing flour at seven different moisture contents, were stored at 72° F. and 100° F. Samples were withdrawn from storage and baked and chemically tested at predetermined intervals. The cakes were judged for quality, and their specific volumes and pH's were determined.

MATERIALS AND METHODS

The varieties of cake mix studied were white, yellow, gingerbread, and devil's food—probably the most popular of the varieties sold in this country. Formulations of the mixes are listed in Table 1.

TABLE 1
Formulations of experimental cake mixes

	White cake	Yellow cake	Devil's Food cake	Gingerbread cake
	%	%	%	%
Flour [1]	36.7	36.0	29.6	33.0
Sugar	43.0	42.1	43.0	37.3
Shortening	12.4	12.3	12.4	12.4
Baking Powder	2.0	2.0	0.6	2.0
Nonfat Milk Solids	3.0	3.0	3.0	3.0
Dry Egg Albumen	2.0	....	....	....
Dry Whole Egg	....	3.6	3.6	3.6
Sodium Bicarbonate	....	....	0.9	....
Salt	1.0	1.0	0.9	0.7
Cocoa	....	....	6.0	....
Molasses Solids	....	....	....	6.9
Spice Mixture	....	....	....	1.1

[1] 14% moisture basis.

Flavoring ingredients which are ordinarily present in small quantities in commercial cake mixes (e.g. vanilla and lemon oil) were not added because it was felt that their presence would not alter the course of staling and yet their odor and flavor might obscure the organoleptic changes taking place in the mix. On the other hand, flavoring ingredients such as molasses solids and cocoa were added because they make a significant contribution to the baking properties as shown by the physical and chemical characteristics of the cake. For example, cocoa can appreciably alter the pH of the product, and molasses solids contain considerable quantities of elements known to accelerate fat oxidation.

A high stability hydrogenated vegetable oil shortening containing 3.2% monoglycerides (as mono-oleate) was used in these mixes. The cocoa was a natural type with a fat content of 25%, a pH of 5.72 and a moisture content of 2.7%. The molasses solids had a moisture content of 1.63%, an ash of 8.72%, reducing sugars (as glucose) 11.9% and sucrose 51.9% ("as is" basis).[d] Chemical characteristics of the flours were: ash—0.33%, protein—7.53%, fat—1.04%, and pH—4.9, all on a 14% moisture basis. In Table 2 are listed other pertinent analytical values for the flours. The differences are due to

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[d] Unless otherwise noted, tests were performed according to the appropriate procedure in AOAC Methods *(1)*.

changes which occurred during the storage period of about 19 months subsequent to drying and prior to incorporation in the mixes.

TABLE 2
Chemical characteristics of dehydrated and control flour

Final flour moisture content	Maximum flour temperature during dehydration	Peroxide value[1]	Carbonyl value[2]	Free fat acidity[3]	Lipid fluorescence[4]
%	° F.				
13.7	......	NF	35	21.4	127
8.8	108	NF	27	11.6	142
7.4	125	NF	24	10.1	147
5.3	140	NF	30	8.5	173
4.7	156	8	37	8.0	164
3.2	183	292	221	8.0	216
2.6	159	209	348	4.4	240

[1] As milliequivalents of peroxide per kilogram of fat.
[2] As micromoles per gram of fat.
[3] As percent oleic acid in the fat.
[4] As microgram equivalents of quinine sulfate per gram of fat.

The egg products used had been desugared by an enzymic process. The dry albumen contained 6.95% moisture and 79.1% protein. The dry whole eggs contained 4.95% moisture, 41.2% fat, 44.3% protein, and 0.48% lipoid phosphoric acid. Less than 0.1% of reducing sugars was found in these egg products.

High-heat non-fat milk solids were used in the mixes. This product contained 2.13% moisture, 1.03% fat, 1.36% titratable acidity (as lactic) and 0.5 mg. iron per 100 g.

Mixes were prepared by beating together all ingredients except the flour and cocoa in the stainless steel bowl of an electric mixer until a "creamed" mass was formed, then adding the remaining ingredients and mixing until the product was homogeneous. Five-hundred gram portions of this mixture were filled into No. 2½ cans which were then sealed immediately. The cans were stored in rooms held at 72° ± 1° F. and 100° ± 1° F. Moisture contents of the mixes are given in Table 3.

Samples for baking tests were withdrawn from 72° F. storage at 6, 12, and 18 months, and from 100° F. storage at 1, 2, 4, 6, 8, 12, and 18 months.

Cakes were prepared according to a standardized baking procedure. An amount of water corresponding to 50% of a standard formula mix and corrected for the difference in flour

TABLE 3
Original moisture content of experimental mixes

Flour	White cake mix	Yellow cake mix	Gingerbread cake mix	Devil's Food cake mix
Moisture content	Moisture content	Moisture content	Moisture content	Moisture content
%	%	%	%	%
13.7	5.1	5.0	4.8	4.3
8.8	3.2	3.4	3.3	2.9
7.4	2.9	2.8	2.9	2.5
5.3	2.2	2.0	2.2	2.0
4.7	1.9	1.8	1.9	1.7
3.2	1.3	1.3	1.6	1.2
2.6	1.2	1.1	1.3	1.1

moisture content so that each batter contained the same ratio of dry matter to moisture, was added in 2 equal portions to the mix while it was being agitated at low speed in a Hobart mixer. Each time, after adding the water, the batter was agitated for 2 minutes at medium speed. After mixing, the specific gravity was determined.

The batter was poured into circular 8-inch aluminum cake pans containing a paper disc on the bottom. The white and gingerbread cakes were scaled at 400 g., the yellow at 375 g. and the devil's food at 350 g. Cakes were baked at about 350° F. Because of difficulty in maintaining an exact temperature while baking a large number of cakes and because of other variables, the cakes were left in the oven until the technologist judged them to be satisfactorily baked. A series of replicate bakes demonstrated that this is a reliable timing method when used by an experienced technologist.

After baking, cakes were allowed to cool, removed from the pan, and prepared for photography. After photographing under uniform conditions, the cakes were weighed and the volume determined by rape-seed displacement. Cakes were sliced vertically and horizontally and scored for texture, grain, crumb color, and odor. The pH was determined by mixing 200 ml. of distilled water with 100 g. of crumb, letting the paste stand for 20 minutes and then testing it on an electronic pH meter.

Chemical analyses of the mix samples stored at 72° and 100° F. were made at 0, 6, 12, and 18 months. Percentage of carbon dioxide in the can headspace was determined as an indication of the extent to which premature reaction of the leavening ingredients had occurred. Moisture contents of the mixes were determined by the vacuum oven method. Fat was extracted from the mixes, and the free fat acidity, peroxide value, and carbonyl value *(5)* were determined. Saline fluorescence was determined by the method of Pearce and Thistle *(8)* modified as to the sample size and method of standardization, and lipid fluorescence was similarily determined using a chloroform extract. The pH was determined on a mix slurry by the electrometric method.

RESULTS AND DISCUSSION

Specific gravities of the batters showed no discernible trend and so are not reproduced here. Slight variations in mixing procedure appeared to have more effect on the specific gravities than did storage time or temperature. Specific volumes of representative test cakes are shown in Table 4. These figures indicate that mixes made with flour of 13.7% moisture content deteriorated greatly during storage for 18 months at 100° F. and finally became wholly unacceptable. All white and yellow mixes made with drier flour tolerated storage for one year at 72° or 100° F. reasonably well, producing cakes which were acceptable (from a volume standpoint) at the end of that time. On the other hand, the gingerbread cake mixes and the devil's food cake mixes made with flour of 8.8% moisture had deteriorated considerably at the end of a year's storage at the

TABLE 4
Specific volumes of cakes made from stored mixes

Mix variety	Moisture content	Storage temperatures 72° F. Storage time (months) 0	6	12	18	100° F. Storage time (months) 1	2	4	6	8	12	18
	%											
White	5.1	3.4	3.2	3.0	2.9	3.0	2.9	2.6	2.4	2.0	1.8	....
White	3.2	3.3	3.2	3.2	3.1	3.1	3.0	3.0	3.2	3.1	3.2	2.8
White	2.9	3.1	3.1	2.8	3.0	2.8	2.8	3.1	3.1	2.9	2.9	2.7
White	1.2	3.4	3.0	3.0	2.9	3.1	3.0	3.0	2.9	2.8	2.5	2.6
Devil's Food	4.3	2.9	2.5	1.6	1.9	2.5	2.3	2.1	2.0	1.8	....	....
Devil's Food	2.9	3.0	2.7	2.5	2.4	3.0	3.1	2.6	2.6	2.4	....	....
Devil's Food	2.5	3.0	3.0	2.8	2.7	3.0	2.6	2.8	2.8	2.9	....	....
Devil's Food	1.1	3.1	3.0	2.6	2.4	3.2	2.8	2.8	2.8	2.7	2.6	2.6

higher temperature. Furthermore, these 2 varieties made with 13.7% moisture flour did not withstand a year's storage at 72° F. Maximum mix stability was secured by using a flour of 7.4% moisture content and additional drying of the flour did not increase mix stability. In fact, there is some evidence that mixes containing flour of 3.2% and 2.6% moisture did not withstand storage as well as mixes containing slightly moister flour, as evidenced by more browning and a somewhat greater decrease in specific volume in the driest mixes. This phenomenon is not clear-cut, however.

Table 5 lists the pH values of crumb slurries made from representative test cakes. These data confirm, to a considerable extent, the trends indicated by the specific volume data. The greatest changes in pH occurred in cakes made from the mixes of highest moisture content. Mixes made from flour of 7.4% moisture content or less remained relatively stable, except in the case of the devil's food mix. Mixes made from flour of 7.4% moisture content and above produced cakes of considerably lower pH after one year storage. All of the pH changes observed were in the direction of greater acidity, except for a few anomalous cases probably explainable on the basis of experimental or sampling error. Furthermore, the more alkaline the cake at the beginning of the storage period, the greater was the decline in pH value.

The changes in pH value are perhaps due to the premature reaction of the baking powder components in the mix. This reaction is also responsible for the drop in cake volume, less CO_2 being available for leavening when the batter was mixed.

In Figures 1 through 4 are illustrated representative cakes made from mixes described in this article. Not all moisture levels are shown, but those omitted to save space fit into the trend established by the illustrated examples. Figure 5 illustrates the trend in a devil's food cake mix of intermediate moisture content.

The main point established by Figures 1 through 5 is that a significant decline in overall quality is associated with extreme changes in pH and volume. An increase in fragility accompanies storage deterioration. In the lighter colored cakes (white and yellow) browning becomes evident. Contours of the devil's food cakes change, the top crust becomes first concave and later flat, while the other varieties become very flat in profile after a somewhat longer storage period. The grain becomes dense, and spots of unassimilated or unhydrated mix appear in the cake. The crumb becomes very soggy.

Mixes containing flour of 7.4% moisture or less were, in the main, resistant to the manifestations of storage deterioration. To be sure, some browning was observed in the white cake mixes of lowest moisture content, but this effect was not nearly as pronounced as it was in the moister mixes of this variety.

In Table 6 are tabulated the headspace carbon dioxide percentages of representative mixes. Headspace carbon dioxide percentages demonstrated quite clearly the

TABLE 6

Percent of carbon dioxide in headspace gases of canned white cake mixes

Moisture content	Storage temperatures						
	72° F.				100° F.		
	Storage time (months)				Storage time (months)		
	0	6	12	18	6	12	18
%							
5.1	2.2	9.8	18.0	24.0	44.9	60.4	72.5
3.2	0.9	3.0	3.6	19.1	8.9	17.5	25.8
2.9	0.6	2.7	2.0	2.7	6.0	11.2	16.6
2.2	0.6	0.8	0.5	0.5	2.7	3.9	5.9
1.9	0.3	0.5	0.5	NF	1.6	3.2	4.7
1.3	0.3	0.4	0.5	0.8	1.7	2.5	3.9
1.2	0.5	0.7	0.8	0.5	1.3	1.6	2.7

increase in rate of carbon dioxide evolution with increased moisture content. There was some reaction of leavening ingredients in all of the mixes, but below certain moisture levels the rate was very slow. The critical level for white cake mixes appears to be between 2.2% and 2.9%, for yellow cake mixes between 1.8 and 2.0%, for gingerbread mixes between 1.6% and 1.9%, and for devil's food cake mixes between 1.1% and 1.2%.

In Table 7 are tabulated the percentages of total carbon dioxide in representative samples of the original and stored mixes. The same trends observed in the headspace CO_2 data are again seen, although the procedure for total carbon dioxide appears to be much more subject to experimental or sampling error. From these data, the leavening ingredients of the mix are seen to be the source of the carbon dioxide found in the can headspace, as expected.

In Table 8 are tabulated representative data from the free fatty acids determinations. The picture of rapid deterioration in the samples of higher moisture content and little or no deterioration in the driest samples is repeated. However, there appear to be no sharp breaks

TABLE 5

pH values of cakes made from stored devil's food cake mixes

Moisture content	Storage temperatures										
	72° F.				100° F.						
	Storage time (months)				Storage time (months)						
	0	6	12	18	1	2	4	6	8	12	18
%											
4.3	9.3	8.9	8.1	8.0	8.8	9.2	8.8	7.7	7.6	7.3	6.8
2.9	9.3	9.0	8.8	8.6	9.0	9.2	9.2	9.2	8.8	8.2	7.8
2.5	9.4	8.9	8.8	8.8	9.0	9.3	9.2	9.1	8.9	8.5	8.1
2.0	9.1	9.0	9.0	8.9	9.0	9.3	9.4	9.0	9.1	9.2	8.7
1.7	9.3	9.0	8.9	8.9	9.0	9.3	9.3	9.1	9.3	9.4	8.8
1.2	9.3	9.1	9.0	8.8	9.0	9.4	9.4	9.3	9.3	9.3	8.7
1.1	9.1	9.0	9.0	8.6	9.2	9.3	9.3	9.2	9.2	9.3	8.7

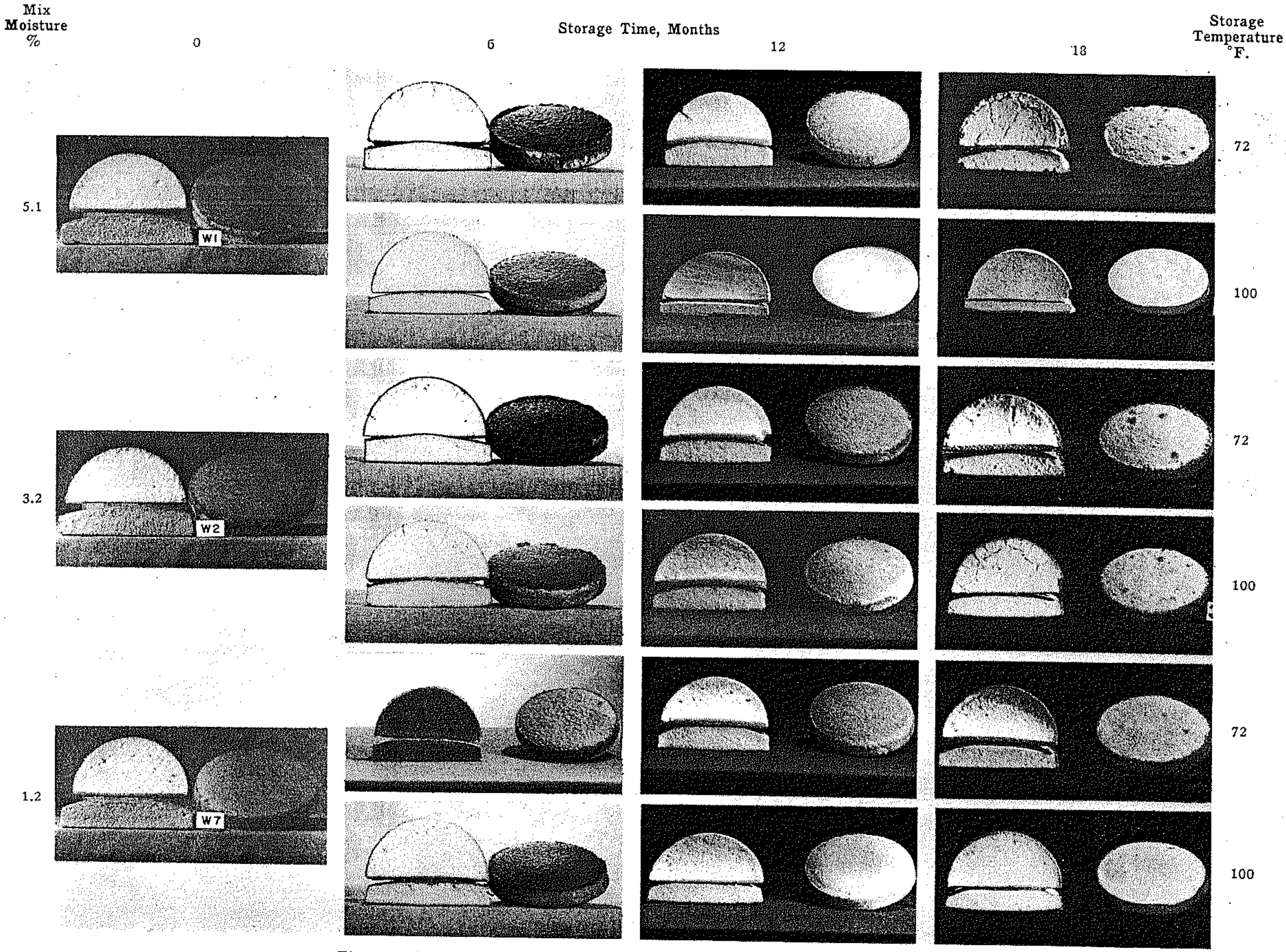

Figure 1. White cakes made from mixes subjected to various storage treatments.

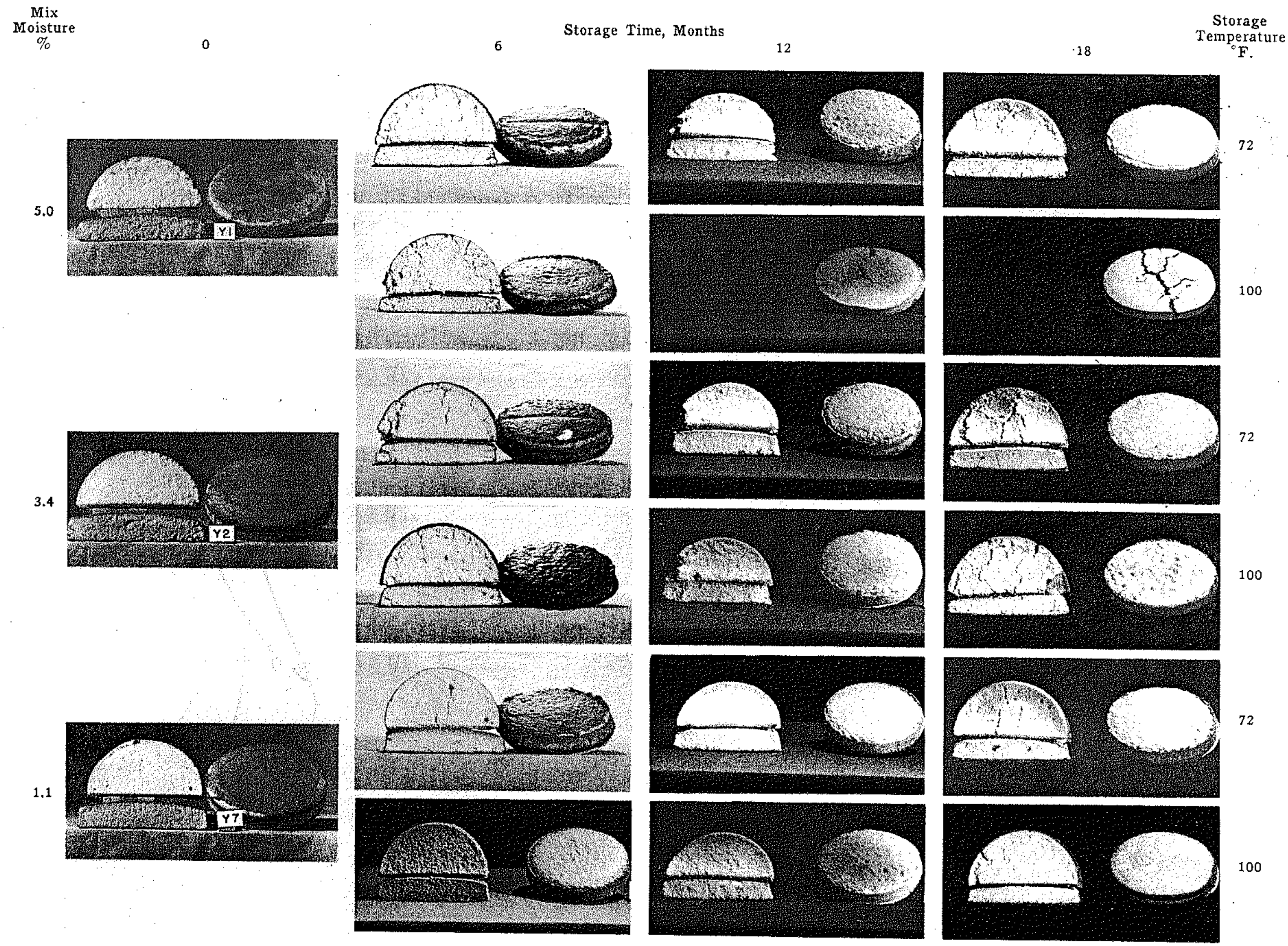

Figure 2. Yellow cakes made from mixes subjected to various storage treatments.

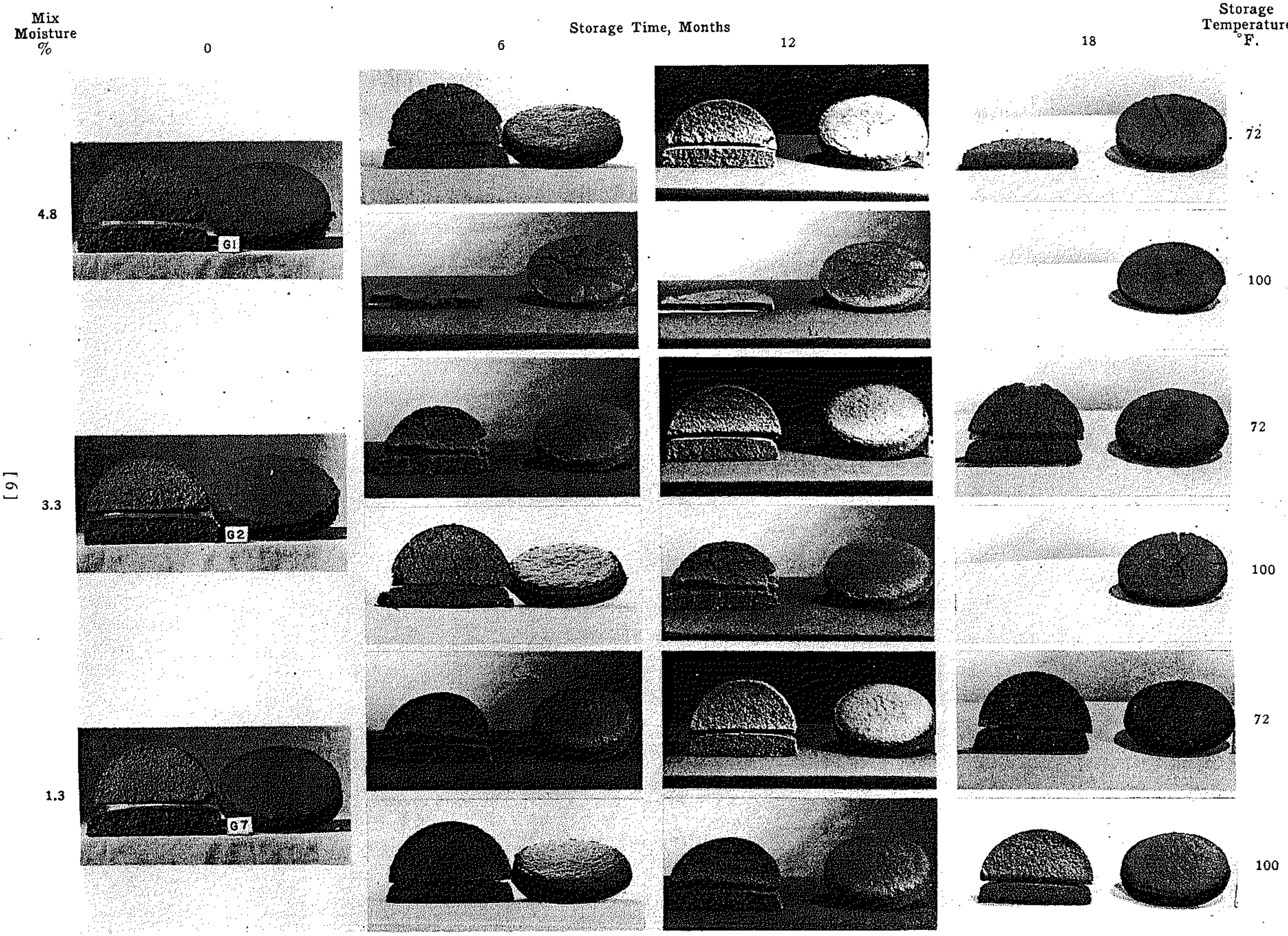

Figure 3. Gingerbread cakes made from mixes subjected to various storage treatments.

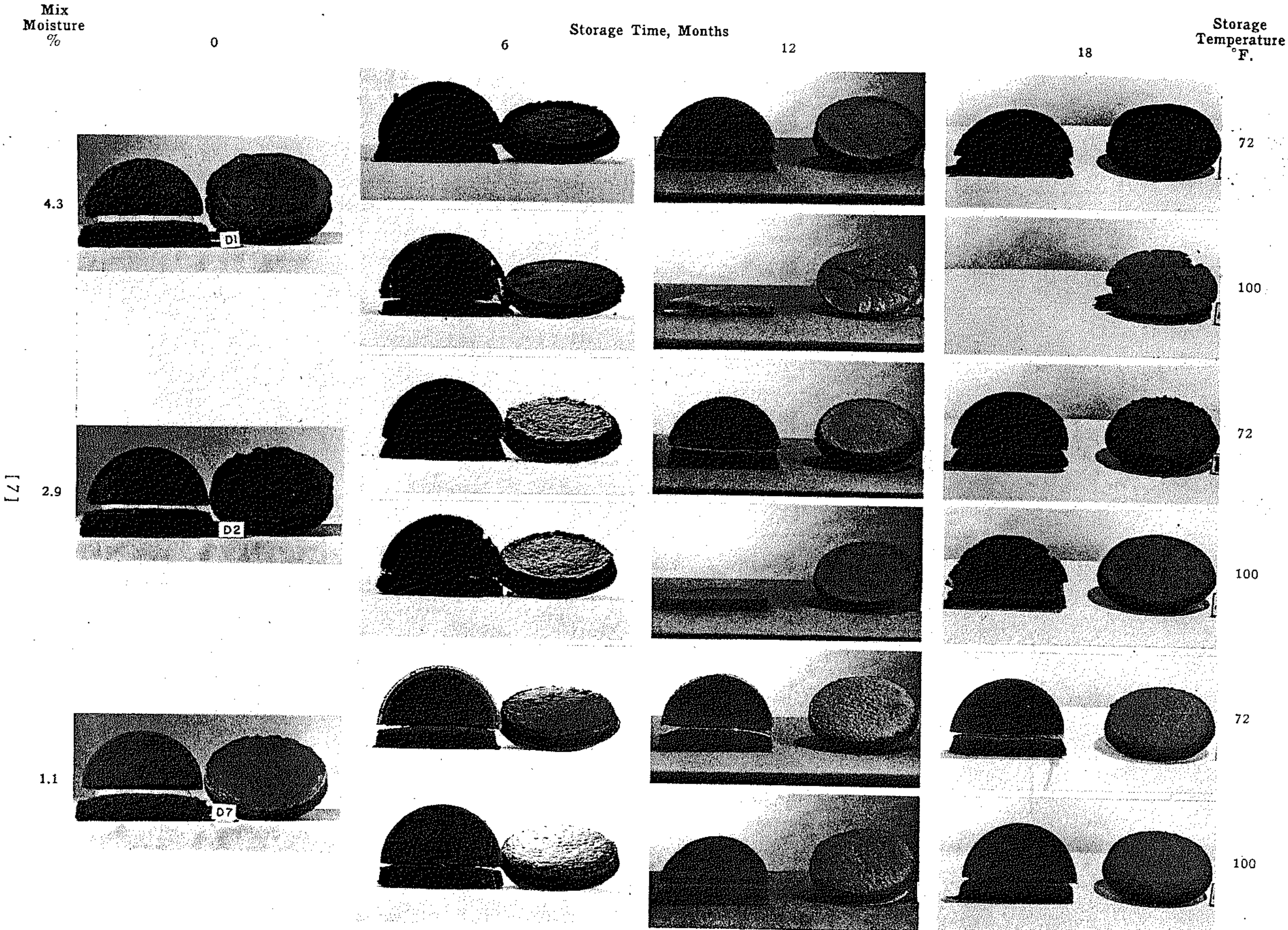

Figure 4. Devil's food cakes made from mixes subjected to various storage treatments.

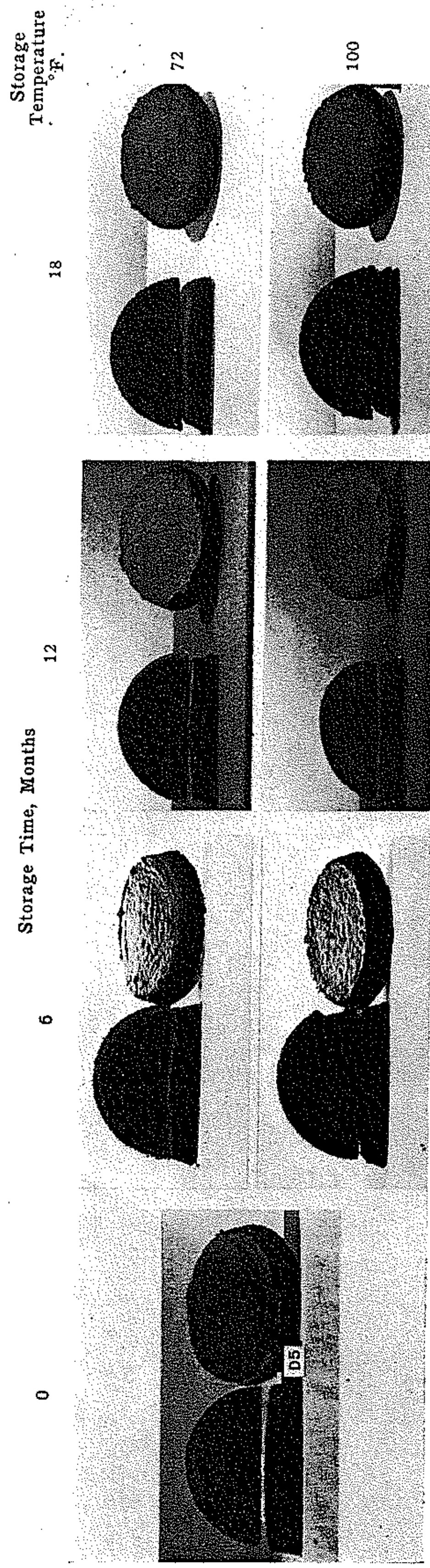

Figure 5. Devil's food cakes made from a mix of 1.7% moisture content stored under various conditions.

indicating a critical level. Rather, there is a gradual decline in free fatty acid production which continues down to the lowest moisture level studied.

Quite a different picture is presented by the data for peroxide values in Table 9. These figures, though rather erratic, show a trend toward faster development of peroxide values in the drier mixes. Little or no increase in peroxides was observed in the 5 mixes of highest moisture content. A considerable increase did occur in the two driest white, devil's food and yellow mixes, but not in the gingerbread mixes. It is interesting that the gingerbread cakes were the lowest in pH of all the varieties. Perhaps this factor has some connection with the absence of peroxide development in the mixes.

Strangely enough, in view of the large fluctuations of pH in the cakes, there were no changes of consequence in the pH values of the mixes themselves. Among those changes which occurred there was no discernible trend.

The carbonyl values also remained relatively constant throughout the period of storage. No changes attributable to deterioration could be discovered. Because of the absence of significant data, the results of these determinations are omitted.

Lipid fluorescence values usually change during storage as a result of reactions of the Maillard type involving phospholipids and sugars or aldehydes, and ending in fluorescent compounds soluble in fat solvents. These values are particularly useful in estimating the amount of staling in products containing egg solids. For these reasons, lipid fluorescence values were determined on the mixes. Changes were erratic, but a gradual increase occurred during the 18 months of storage. The increase appeared to be somewhat accelerated by lower moisture contents and higher temperatures. For example, the white cake mix of lowest moisture content had a lipid fluorescence of 8.3 (microgram equivalents of quinine sulfate per gram of fat) at the start and 8.6 after 18 months at 72° F. and 10.0 after 18 months at 100° F. Equivalent figures for the devil's food cake mix of highest moisture content are 8.4, 14.8, and 14.0, respectively. Other data are omitted to save space.

An increase in saline fluorescence values during storage was expected but these saline fluorescence values showed little change and, for that reason, are not tabulated here. There was a slight increase in saline fluorescence values of the wetter mixtures and, apparently, a slight decrease in the drier mixtures over the total storage period. The temperature of storage apparently had little influence on the rate at which these changes occurred, and the random fluctuations in these analytical values were so large as to render the existence of any significant trend obscure.

SUMMARY

It has been shown that the rate of deterioration of chemically leavened cake mixes during storage at ordinary or relatively high temperatures is dependent upon the moisture content of the mix and the variety of the mix. Products formulated with flour of 7.4% moisture content, or less, were generally quite resistant to deleterious aging reactions. Mixes producing cakes having

TABLE 7

Effect of storage on total carbon dioxide in cake mixes

Mix variety	Moisture content	Storage temperatures						
		72° F.				100° F.		
		Storage time (months)				Storage time (months)		
		0	6	12	18	6	12	18
	%	%	%	%	%	%	%	%
White	5.1	0.59	0.25	0.16	0.16	0.12	0.05	0.05
White	3.2	0.30	0.30	0.25	0.24	0.31	0.22	0.26
Yellow	5.0	0.31	0.26	0.19	0.19	0.09	0.03	0.04
Yellow	3.4	0.19	0.32	0.25	0.26	0.26	0.19	0.14
Yellow	2.8	0.29	0.32	0.24	0.28	0.29	0.22	0.22
Gingerbread	4.8	0.29	0.23	0.16	0.14	0.09	0.04	0.05
Gingerbread	3.3	0.32	0.29	0.24	0.29	0.26	0.15	0.12
Gingerbread	2.9	0.30	0.30	0.27	0.30	0.30	0.29	0.22
Devil's Food	4.3	0.44	0.43	0.30	0.30	0.22	0.08	0.06
Devil's Food	2.9	0.48	0.51	0.39	0.46	0.42	0.28	0.26
Devil's Food	2.5	0.46	0.51	0.32	0.33	0.46	0.32	0.32

TABLE 8

Free fatty acids in the fat of stored cake mixes

Mix variety	Moisture content	Storage temperatures						
		72° F.				100° F.		
		Storage time (months)				Storage time (months)		
		0	6	12	18	6	12	18
	%	%	%	%	%	%	%	%
White	5.1	0.72	1.28	2.60	3.89	3.96	14.77	22.34
White	3.2	0.37	0.47	0.61	1.58	0.75	1.55	2.08
White	2.9	0.37	0.43	0.53	0.62	0.57	0.92	1.34
White	2.2	0.32	0.33	0.36	0.39	0.36	0.48	0.60
White	1.9	0.24	0.31	0.35	0.36	0.31	0.41	0.49
White	1.3	0.25	0.28	0.30	0.30	0.26	0.30	0.34
White	1.2	0.21	0.27	0.28	0.28	0.22	0.26	0.30
Devil's Food	4.3	1.05	2.22	3.64	4.20	4.54	11.15	17.72
Devil's Food	2.9	0.81	1.06	1.60	1.96	1.59	3.66	3.34
Devil's Food	2.5	0.83	0.90	1.24	1.44	1.27	2.67	2.60
Devil's Food	2.0	0.84	0.78	0.94	1.06	0.92	1.63	1.74
Devil's Food	1.7	0.79	0.77	0.83	0.90	0.75	1.06	1.21
Devil's Food	1.2	0.74	0.67	0.66	0.70	0.67	0.71	0.80
Devil's Food	1.1	0.67	0.70	0.82	0.68	0.65	0.62	0.71

TABLE 9

Peroxide values of fat of stored cake mixes[1]

Mix variety	Moisture content	Storage temperatures						
		72° F.				100° F.		
		Storage time (months)				Storage time (months)		
		0	6	12	18	6	12	18
	%	%	%	%	%	%	%	%
White	5.1	NF	3.1	4.9	1.8	4.7	2.5	NF
White	3.2	NF	2.5	3.4	1.9	4.7	3.1	NF
White	2.9	NF	2.3	2.7	2.0	3.4	3.6	NF
White	2.2	NF	4.0	2.6	1.8	4.3	7.6	20.6
White	1.9	NF	2.0	3.2	7.2	3.8	20.2	33.0
White	1.3	13.0	15.7	23.0	24.1	5.7	35.1	36.2
White	1.2	11.0	10.9	14.5	15.9	15.4	20.7	19.8
Gingerbread	4.8	NF	1.7	1.8	0.8	0.4	2.0	1.8
Gingerbread	3.3	NF	0.6	2.7	2.0	0.6	1.6	0.4
Gingerbread	2.9	NF	0.5	2.0	1.5	1.0	3.2	2.4
Gingerbread	2.2	NF	4.0	2.6	1.7	2.4	2.6	4.3
Gingerbread	1.9	NF	4.2	2.3	2.2	0.2	3.7	3.8
Gingerbread	1.6	NF	4.4	2.9	3.0	0.9	2.2	3.7
Gingerbread	1.3	6.3	3.8	2.6	4.4	1.7	2.9	4.1

[1] Milliequivalents per gram of fat.

crumb of nearly neutral pH were more stable than mixes producing cakes having more alkaline crumb.

Changes in the physical properties of cakes resulting from storage of the mixes are due primarily to a premature reaction of the leavening ingredients. Carbon dioxide normally available during the baking period is lost as a result of this reaction, and substances having a weakening effect on the gluten structure are accumulated in the aged mix. The final result is a cake of low specific volume, altered pH, and soggy and fragile texture.

Another change occurring in stored mixes of moderate to high moisture contents is an increase in free fatty acids. On the other hand, a considerable increase in peroxide value of the ether extractables is found in the drier mixes, while the peroxide values of higher moisture content mixes are nearly unaffected by storage.

Higher moisture contents appear to accelerate slightly the increase in saline fluorescence during storage, whereas the opposite is true for lipid fluorescence. These changes are not extreme, however.

Factors apparently unaffected by storage under the conditions employed in this experiment are pH of the mixes and their carbonyl values.

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