

wheats including the blends and all soft wheats including soft wheat blends is presented in Table IV. There were only three statistically significant differences. Hard wheats were higher than soft wheats in Fe and Zn, while soft wheats contained significantly more K than hard wheats.

d. Correlation Coefficients between Protein and Ash Content and Mineral Elements. Correlation coefficients between percent protein and percent ash in wheats and each of the eight mineral elements were calculated for all hard wheats and hard wheat blends, for all soft wheats and soft wheat blends, and for all wheat classes combined. The correlation coefficients are given in Table V.

A significant positive correlation was established between the protein content of hard wheats and hard wheat blends and Ca, Fe, K, and Cu content and between ash content of those wheats and Ca, Mg, Na, K, Na, Mn, and Cu.

In soft wheat and soft wheat blends, significant positive correlation was found between percent protein and Ca and Zn contents. Soft wheat ash was also significantly correlated with Zn, Mn, and Cu content in soft wheat.

Combining all classes of wheat, it can be seen from the values in Table V that wheat protein is significantly correlated with Ca, Zn, Fe, and K content, while percent ash shows a significant correlation with Ca, Mg, Zn, K, Na, and Mn.

A full discussion must include application of these data. Specifically, it is clear that the classes of hard and soft wheats analyzed show a number of significant differences in terms of naturally occurring mineral contents. If the intended cereal fortification program discussed in the introduction is to take place, selection of proposed fortification levels must take these naturally occurring differences into account. It must be realized that, although

"typical", or average, values for these minerals may be convenient for textbook discussion, a large-scale fortification program, ultimately coming under government compliance regulations, will require a knowledge of the wide range of differences between wheat classes shown in this study and those to come.

ACKNOWLEDGMENT

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Effects of 1,3-Diols and Their Esters on the Rheological Properties of Dough and the Storage Stability of Bread

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Several linear 1,3 diols and their monoesters were studied in standard bread formulations. The lower 1,3-diols, especially 1,3-butanediol, exhibited antistaling properties while the higher diols were effective mold inhibitors. The esters, in amounts ranging from 0.1 to 0.5%, improved the mixing characteristics of test doughs, inhibited mold formation, and, in some instances, increased loaf volume and enhanced bread quality. The most effective esters were those whose total carbon content, diol plus acid portion, was in the range of C₁₁-C₁₂. The potential of these compounds as functional bread additives and problems to be overcome are discussed.

A variety of additives have been used as processing aids for bread doughs and other baked goods. Such materials are normally added to improve one or more of the following characteristics: mixing tolerance, dough strength, bread volume, texture, or softness. In addition, chemical ad-

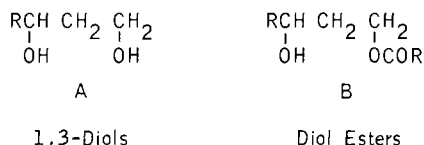
ditives are often employed to retard mold spoilage and thereby increase the shelf life. Among the additives used or recommended as dough conditioners are salts of steryl lactic acid (Marnett and Tenney, 1961; Tenney and Schmidt, 1968), polypropylene glycol (Moneymaker and Forsythe, 1974), glycosides of hydroxy fatty acids (Bauerlen and Findley, 1969), glycerides of succinic acid (Meisner, 1969), and sulfosuccinates (Whelan, 1970). Softening agents and volume improvers are usually monoglycerides (Church, 1973) although other emulsifiers may be used including the lactylate and sucrose derivatives (Pomeranz and Finney, 1973). Pomeranz and Wehrli (1969) also recommend synthetic glycosylglycerides. Salts of propionic acid or sorbic acid are the most common mold

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inhibitors (Pomeranz, 1969).

As part of our search for new foods and food additives we have found that certain linear 1,3-diols and their esters are potentially useful multipurpose additives for baked goods (Frankenfeld et al., 1972). These materials, which have the general chemical structures A and B are readily



metabolizable, energy dense materials which actually serve to improve the nutritional qualities of the products to which they are added (Frankenfeld et al., 1975; Frankenfeld and Miller, 1974). The results of acute toxicity and metabolic studies of the diols and esters have been described in detail elsewhere (Frankenfeld and Miller, 1974; Frankenfeld et al., 1975; Giron, 1968). Briefly, it was found that of all isomeric dialcohols and esters of dialcohols the linear 1,3 isomers are uniquely nontoxic (LD_{50} 's > 20 g/kg in rats) and readily metabolized.

MATERIALS AND METHODS

Test Chemicals. The 1,3-diols used in this study were prepared by reduction of the corresponding hydroxy esters (Frankenfeld and Werner, 1969) with lithium aluminum hydride in ether (Gaylord, 1956). The esters were prepared by reacting the appropriate diols with acid chlorides in ether. All test compounds were purified by vacuum distillation. Purity of 99.5% or better was verified by gas chromatography. Food grade softening agents and bread preservatives, which were used for purposes of comparison, were obtained from commercial sources and were used without purification.

Baking Trials. Two types of experiments were conducted. In order to determine the rheological properties of doughs containing the diols and esters and to obtain preliminary data on bread quality, the following procedure was followed: (1) A "sponge" was prepared by mixing the following ingredients (only one type of flour was used): flour, 225 g; salt (NaCl), 0.3 g; yeast food (NH_4Cl), 0.15 g; baker's yeast, 7.15 g; water, 100 g.

All of the above ingredients were mixed for 2 min in the mixing bowl of a standard experimental mixer (Farinograph) equipped with instruments for measuring and recording of resistance of the dough of mixing (Stafford, 1970; Kent-Jones and Amos, 1967).

(2) After mixing, the "sponge" was allowed to ferment for 3.5 h, at 86 °F.

(3) After fermentation the sponge was placed in the mixing bowl, and the following ingredients were added: flour, 75 g; salt, 5.7 g; sugar (sucrose), 18 g; nonfat milk solids, 12 g. These were mixed for 0.5 min.

(4) Water (50 cm³) was then added. When additives were used they were dissolved in this water to form a solution, or dispersion, depending on additive solubility. The additives that were used were at 1.5 g (0.5% level) or 3.0 g (1% level) per total weight of flour. Control loaves containing no additives were prepared in the same manner.

(5) The dough was then mixed at low speed for an additional 1.5 min at a constant temperature of 95 °F.

(6) Mixing was stopped and 9 g of hydrogenated vegetable shortening was added.

(7) The mixer was started at high speed, and after 2–3 min additional water was added to give a standardized resistance to mixing. The amount added was designed to give a maximum consistency of 500 Brabender units as measured with a Farinograph. Using this amount as a

guide, an additional dough was prepared and a typical Farinograph and Extensigraph curve was prepared to evaluate typical flour dough parameters (Stafford, 1970; Kent-Jones and Amos, 1967). Each dough formulation was tested in triplicate. A constant temperature of 95 °F was maintained during mixing.

For the preparation of breads mixing was continued for 3 min past the time at which maximum consistency was reached. Typically, a total mixing time of 12 min resulted.

(8) The dough was taken from the mixing bowl and a second fermentation at 86 °F, 98% RH (relative humidity) was carried out for 1 h.

(9) The dough was divided into 150-g portions and rolled into a ball. This was rested for 8 min. Pup loaves were then formed using the Brabender Extensigraph (Stafford, 1970; Kent-Jones and Amos, 1967).

(10) The loaves were then placed in greased pup pans and proofed to constant height of $\frac{3}{8}$ in. above the pan line in a box maintained at 97 to 98 °F at 80 to 90% relative humidity.

(11) After proofing the loaves were baked in a rotary brick oven at 425 °F. The baking time was 20 min.

The loaves thus obtained were evaluated for freshness (antistaling properties) during storage. Loaves with various additives were compared against controls. The degree of staling was determined by measuring the depth of penetration by a plunger into slices of test loaves when known weights were placed on the plunger (Platt and Powers, 1940; Bice and Geddes, 1949). The depth was measured with a cathetometer. The degree of penetration was considered to be a measure of bread softness. Measurements were made on six–eight slices taken from the center of each loaf. The results were averaged and the standard deviation and standard errors were calculated.

Both control loaves and those containing the test compounds were evaluated for bread quality using the American Institute of Baking standard scoring method (Matz, 1960). In addition, both whole and sliced loaves were bagged, sealed in metal containers, and stored under ambient conditions. The loaves were examined daily for the appearance of mold.

In order to evaluate the test compounds for antistaling and preservative properties under commercial bread-making conditions, a slightly different procedure was followed (Shukis, 1970).

A commercial sponge-dough formula was used incorporating 60% of the total flour in the sponge. Standard amounts of dry milk solids, sugar, salt, and shortening were used in all doughs. Each series consisted of four doughs each of which yielded six loaves.

One loaf was bagged unsliced and another sliced and bagged and put aside for mold detection. Of the remaining loaves one was used for scoring and the remaining were used for antistaling evaluation.

The first dough of each series was a "no additive" control. The second dough contained a standard amount of 0.5% Atmul 500 (a bread softener), and 0.3125% of calcium propionate (a mold inhibitor). The remaining two doughs contained the test additives in various amounts or combinations to replace Atmul 500 and calcium propionate.

Procedures in mixing, make-up, and baking were standardized according to accepted commercial procedures (Shukis, 1970). The proof time was variable depending upon the time required for the loaves to proof to a height $\frac{5}{8}$ in. above the pan line. The proof time was noted in all cases.

Table I. Effects of Selected Diol Esters on the Rheological Properties of Dough

Additive	Mixing properties (Farinograph test)			Tensile properties (Extensograph test)	
	Stability min	Mixing tolerance index, Brabender units	20-Min drop, Brabender units	"Energy" area under Extensogram, arbitrary units	Resistance to extension ratio
	Additive level: 0.5 g/100 g of flour				
None	10.0	38	60	21.9	1.26
1,3-Butanediol 1-monopropionate	10.3	30	60	26.8	2.16
1,3-Butanediol 1-monooctanoate	18.0	12	20	30.7	1.68
1,3-Butanediol monopalmitate	16.0	20	30	27.3	1.68
1,3-Heptanediol monooctanoate	14.0	30	35	26.7	1.45
1,3-Heptanediol monopalmitate	11.0	38	50	24.0	1.15
1,3-Octanediol 1-monopropionate	17.5	15	15	28.2	2.02
	Additive level: 0.2 g/100 g of flour				
None ^a	7.8	N ^b	60	19.2	1.86
1,3-Butanediol 1-monopropionate	10.5	N	40	17.2	2.66
1,3-Heptanediol 1-monooctanoate	10.4	N	40	17.7	2.58
1,3-Octanediol 1-monopropionate	11.9	N	40	18.5	2.72

^a Separate test requiring new set of controls. ^b N = not determined in this series.

Table II. Effects of 1,3-Diols on Bread Quality

Additive	Overall score (100 maximum) ^a	Average bulk (density), g/cm ³	Average bulk volume, in. ³ /oz
None	88.5	0.204	8.48
1,3-Butanediol (0.5% of flour weight)	92.5	0.206	8.40
1,3-Heptanediol (0.5% of flour weight)	90.0	0.208	8.33

^a American Baking Institute standard scoring method (Matz, 1960).

The test loaves were evaluated for overall score by standard procedures (Shukis, 1970). Antistaling effects were determined by measuring the compressibility of test loaves at 18, 48, 72, and 120 h. The sliced and unsliced loaves were examined for mold infestation each day from 7 days until the termination of the test (13 days).

RESULTS AND DISCUSSION

Effects of Test Compounds on Rheological Properties of Dough. The 1,3-diols tested had no effect on the rheological properties of doughs. On the other hand, certain of their esters were quite effective in improving mixing properties. The results of Farinograph and Extensigraph tests with selected esters as dough additives are summarized in Table I. Of particular importance are the increases in "stability time" and decrease in "mixing tolerance index". The changes indicate improved tolerance of the dough to processing abuses. Examples of the improvement found are illustrated by the Farinograph curves in Figure 1. Shown are the traces for the two best compounds (curves C and D) and one of the poorer ones (curve B) as well as the no-additive control. The increase

in stability of the doughs in the series A-D is apparent. The best compounds tested were 1,3-butanediol 1-monooctanoate and 1,3-octanediol 1-monopropionate. Doughs containing all the materials tested showed improvements in the test parameters at the 0.5% level of addition (i.e., all deviations from controls shown on Table I are in a desirable direction). The best two compounds also imparted improved tensile properties to the doughs (high values for both the "energy" and resistance to extension ratio in the Extensigraph test). At the 0.2% addition level, the doughs containing test compounds showed improvements in all but "energy" as determined by the Extensigraph test. This was about equal to controls for 1,3-octanediol 1-monopropionate but slightly lower in the case of the other esters.

It is interesting that the best additives are those with a short carbon chain in either the diol or ester moiety combined with a medium to long carbon chain in the other portion of the molecule. Thus, the two most promising compounds, 1,3-butanediol 1-monooctanoate and 1,3-octanediol 1-monopropionate, have carbon numbers of 12 and 11, respectively. This effect is undoubtedly related to the hydrophile-lipophile balance (Pomeranz et al., 1970; Ofelt et al., 1958; Meisner, 1969) which is apparently optimum in this range for these types of additives.

Effects of Diols and Esters on Bread Quality and Shelf Life. In concentrations up to 0.5% neither 1,3-butanediol nor 1,3-heptanediol, had any noticeable effects on bread quality. Volume, density, and overall score were essentially the same as controls (Table II). However, these compounds were effective, in different ways, in prolonging the shelf life of bread. The 1,3-butanediol showed significant antistaling properties as evidenced by the increased penetration of the standard plunger (Table III)

Table III. Effects of 1,3-Diols on Shelf Life of Bread

Additive	Penetration of plunger of specified weight into bread after 4 days of storage, cm ^a						Days to first appearance of mold
	400 g of plunger			600 g of plunger			
	Mean	SD	SE	Mean	SD	SE	
None	0.138	0.045	0.016	0.244	0.080	0.027	6
1,3-Butanediol (0.5 g/100 g of flour)	0.226	0.038	0.014	0.314	0.069	0.024	6
1,3-Heptanediol (0.5 g/100 g of flour)	0.195	0.041	0.014	0.277	0.044	0.015	11
1,3-Heptanediol (1.0 g/100 g of flour)	NT ^b			NT ^b			>6 weeks

^a Penetration test: the greater the penetration the softer the bread. ^b NT = "not tested".

Table IV. Effects of Diol Esters on Bread Quality^a

Additive	Proof time, min	Overall score (100 maximum) ^b	Average volume, cm ³	Average specific volume, in. ³ /oz
		Additive level: 0.5 g/100 g of flour		
None	60	88.5	550	7.4
1,3-Octanediol 1-monopropionate	150	88.0	541	7.2
1,3-Butanediol 1-monooctanoate	240	71.0	300	4.0
		Additive level: 0.3 g/100 g of flour		
None	56	86.0	656	8.3
1,3-Octanediol 1-monopropionate	72	92.0	630	8.0
1,3-Butanediol 1-monooctanoate	158	76.5	548	6.8
		Additive level: 0.2 g/100 g of flour ^c		
None	86	90.0	596	7.7
"Atmul 500" (0.5 g/100 g of flour) ^d	79	93.0	658	8.2
1,3-Octanediol 1-monopropionate	109	92.0	626	7.8
1,3-Butanediol 1-monopropionate	150	81.0	371	4.6
		Additive level: 0.1 g/100 g of flour ^c		
1,3-Butanediol 1-monopropionate	72	94.5	661	8.5

^a ND = "not determined". ^b American Baking Institute standard scoring method (Matz, 1960). ^c 0.1% 1,3-nonanediol added as mold inhibitor; apparently had no effect on quality. ^d Commercial bread improver added at usual level (Shukis, 1970).

Table V. Antistaling and Antimycotic Properties of Diol Esters

	Additive level added, g/100 g of flour				
	1,3-Octanediol 1-monopropionate			1,3-Butanediol 1-monooctanoate	
	None	0.5	0.3	0.5	0.3
Penetrability (arbitrary units) ^a					
1 day	0.635	0.528	0.593	0.093	0.397
3 days	0.211	0.401	0.385	0.105	0.168
Drop in penetrability (2 days)	0.424	0.127	0.208	<i>b</i>	0.229
% drop in penetrability (2 days)	67	24	35	<i>b</i>	58
Days to first evidence of mold	6	11	8 ^c	6	6

^a The greater the penetration, the softer the loaf. ^b Poor loaf volume; test not valid. ^c Only very light mold infestation was observed at this point.

Table VI. Results of Commercial Scale Baking Trials

Test additives ^a	Proof time, min	Bread score ^b	Volume, cm ³	Compressibility, g/4 mm ^c			Days to first appearance of mold
				18 h	72 h	120 h	
1. None	50	93.5	2625	79.0	139.9	169.2	<10
"Atmul 500" plus calcium propionate	55	91.5	2650	72.0	120.1	150.4	13
1,3-Butanediol	55	88.0	2700	70.0	107.0	152.9	13
2. None	50	94.0	2700	80.5	132.2	167.1	>8<11
"Atmul 500" plus calcium propionate	63	91.5	2700	75.9	119.4	134.1	>11
1,3-Octanediol 1-monopropionate	75	92.0	2800	86.1	145.5	171.1	11 (d)
3. None	50	94.0	2525	93.7	155.9	185.4	>7<10
"Atmul 500" plus calcium propionate	70	95.5	2550	103.5	134.7	179.0	>10
1,3-Butanediol plus 1,3-heptanediol ^a	70	90.0	2550	102.9	147.6	187.8	>10 ^d

^a All were added at 0.5 g/100 g of dough except calcium propionate which was added at 0.3125% and 1,3-heptanediol which was added at 0.2%. ^b American Bakers Cooperative Inc. standard scoring method (Shukis, 1970). ^c Note that this test is different from those shown in Tables III and V. In this test the lower the value the softer the loaf. ^d Only partial growth at day 24.

and 1,3-heptanediol was effective as a mold inhibitor. At the 1% level the latter additive kept test loaves mold free for over 6 weeks. However, at this level, the bread quality was adversely affected. This diol was effective as a mold inhibitor at levels as low as 0.2%. Although 1,3-heptanediol showed some antistaling properties, it was not so effective as 1,3-butanediol (Table III).

At levels of 0.3% or less most diol esters improved bread quality over controls (Table IV). At 0.2%, 1,3-octanediol 1-monopropionate gave bread scores and loaf volumes comparable with those of a standard commercial emulsifier "Atmul 500" added at 0.5%. It should be noted that 1,3-octanediol 1-monopropionate also shows good dough conditioning properties at the 0.2% level (Table I). The best results in these tests were obtained with 1,3-butanediol 1-monopropionate added at 0.1%. These loaves had a larger volume and overall score than any of the

others tested (Table IV). However, 1,3-butanediol 1-monopropionate is not one of the best dough conditioners (Table I). In amounts exceeding 0.3% the esters tended to have adverse effects on bread quality. This was due to the prolonged proofing time and poor loaf volumes encountered at the higher levels.

One of the esters, 1,3-octanediol 1-monopropionate, exhibited moderate antistaling and antimycotic properties in laboratory tests (Table V). The other ester tested, 1,3-butanediol 1-monooctanoate, showed no improvement over controls in either test. This was due, at least in part, to the poor loaf volumes obtained with this compound. In test tube experiments, the two esters showed comparable antimicrobial activity (Frankenfeld et al., 1973).

Results of Commercial Baking Tests. These were undertaken in order to verify the laboratory observations under conditions comparable with actual commercial

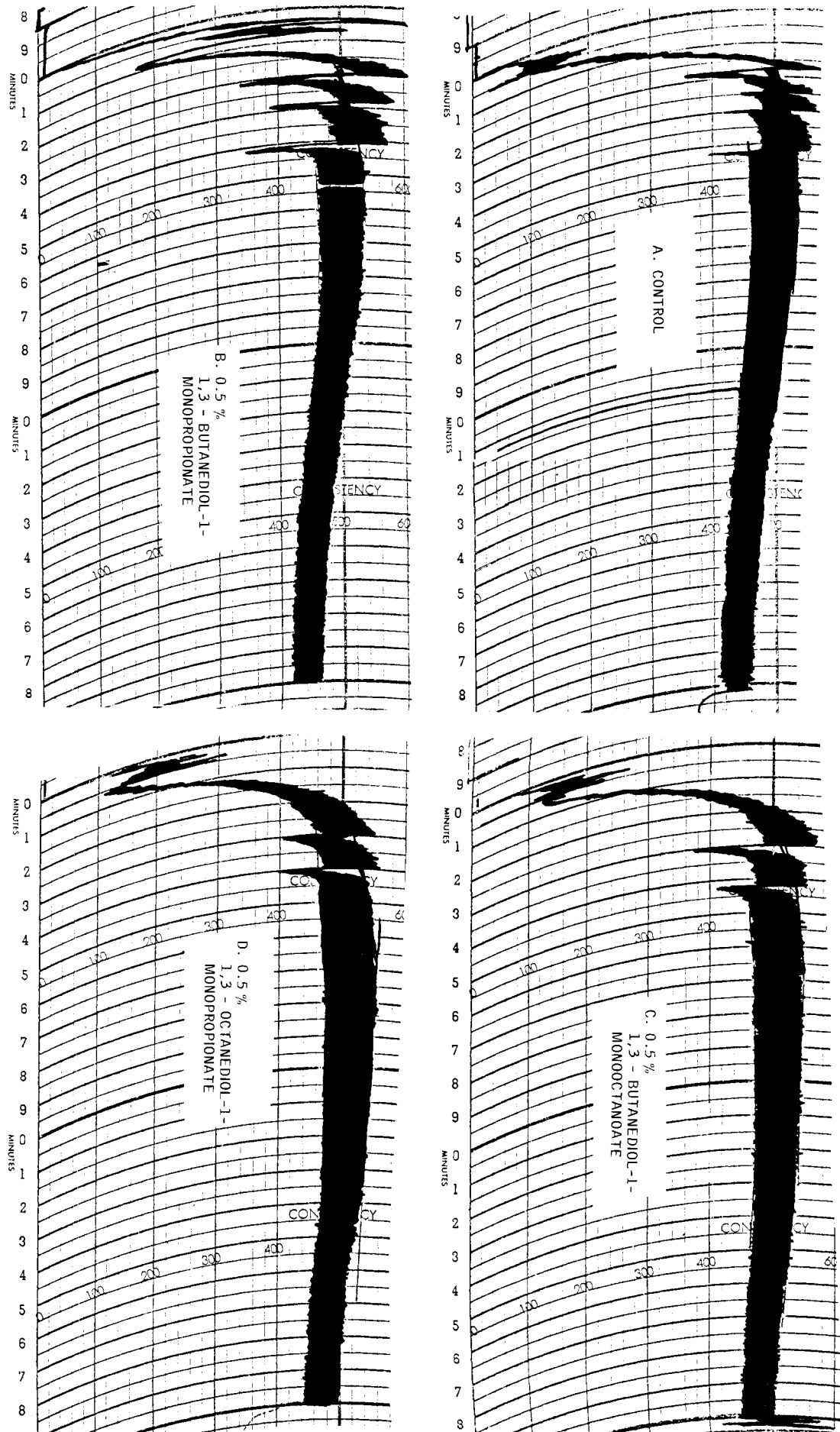


Figure 1. Effects of diol esters on Farinograph mixing characteristics.

baking operations. The effects of selected additives on bread quality (overall score), compressibility, and mold inhibition were investigated. A common commercial bread improver ("Atmul 500" plus calcium propionate) was included to serve as a positive control. The results are summarized in Table VI. These trials confirmed the antistaling properties of 1,3-butanediol and the mycostatic effects of 1,3-heptanediol and 1,3-octanediol 1-mono-octanoate. On the other hand, the ester did not inhibit staling in these tests when employed at the 0.5% level. Lower levels were not tested. Compared with the standard softener, Atmul 500, 1,3-butanediol was significantly more effective at 72 h but about comparable at 120 h storage time. Both the ester, 1,3-octanediol 1-monopropionate, and the diol, 1,3-heptanediol, inhibited mold growth better than calcium propionate. The diol was more effective at 0.2% than calcium propionate at 0.3215%. However, 1,3-heptanediol promoted long proofing times and had an adverse effect on bread softness. Loaf volume was improved by 1,3-octanediol 1-monopropionate and, to a lesser extent, by 1,3-butanediol. Only the loaves containing 1,3-octanediol 1-monopropionate gave a better overall score than those with the Atmul 500-calcium propionate combination. The diol and diol ester loaves were all penalized for "odors not normal to bread". Results in our laboratory and elsewhere (Celanese Corporation, 1970) suggest these may be due to trace impurities or decomposition products produced during synthesis and purification. Techniques for minimizing these have been described (Celanese Corporation, 1970).

CONCLUSIONS

These preliminary results indicate that certain linear 1,3-diols and their monoesters are potential multipurpose bread additives. A major advantage to these materials is their inherent safety. Effectiveness varies in a consistent manner with chemical structure. The best compounds for promoting bread softness are the more hygroscopic diols with carbon numbers of six or less. The most effective mycostats are the diols with carbon numbers ranging from seven-nine and the diol esters whose total carbon content, diol plus acid moieties, ranges from C₁₀-C₁₅ (Frankenfeld et al., 1975). These latter compounds are also those showing the greatest promise as dough conditioners. The major difficulty in exploiting these materials as functional bread additives in overcoming odor problems and the tendency to prolonged proofing times. Additional research is required to improve synthesis procedures, develop new dough formulations and to explore combinations of compounds which yield optimum results at the lowest possible levels of addition. Initial tests on acute toxicity and metabolic fate of the compounds indicated no potential safety problems. However, sufficient data to justify

FDA approval for the test compounds are still to be developed.

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