Effects of Fumigation on Wheat in Storage. IV. Tocopherols

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ABSTRACT

The effects of fumigation and storage on the tocopherols in wheat grain, milling fractions, and baked products were investigated. Wheats were fumigated periodically with methyl bromide, ethylene dichloride/carbon tetrachloride, and phosphine over a 3-year period. The amounts of α -tocopherol, α -tocotrienol, β -tocopherol, and β -tocotrienol, as determined by gas chromatography, did not change significantly during this time as a result of fumigation or storage; storage caused minor losses. Bread and rolls made with the flour were analyzed before and after baking. The retention of tocopherols following baking was high: Only losses in β -tocotrienol were found.

Wheat is commonly stored for two or more years before use. To control insect infestation during this time it is customary to fumigate the storage bins periodically with pesticides. Little is known about the lasting effects of fumigation on either the grain or the products. A multifaceted investigation has been carried out by the U.S. Department of Agriculture in an effort to answer some of the more important questions concerning pesticide retention in the grain, changes in its nutrient content, and alteration of its baking properties. The results of physical testing, experimental baking, and vitamin B-6 analyses have already been published (1,2,3). This paper is concerned with the tocopherol (vitamin E) content of stored fumigated wheat, its milling fractions, and experimentally baked products.

MATERIALS AND METHODS

Kansas-grown Early Triumph wheat with no previous history of fumigation was divided into five lots: two for controls and three for fumigation. The details of storage, fumigation, sampling, and milling have been published elsewhere (1). In brief, fumigant treatments were applied eight times over a 3-year period. The fumigants tested were: 1) methyl bromide, at the rate of 2.5 lb. per 1,000 cu. ft. of storage; 2) a mixture of ethylene dichloride and carbon tetrachloride (CCl₄) (3/1), at the rate of 6 gal. per 1,000 bu.; and 3) phosphine, at the rate of six aluminum phosphide tablets per ton. Two controls were used, one at ambient temperature and one at 32°F. The ambient-temperature control and the fumigated wheats were stored in wooden bins; the refrigerated control at 32°F. was stored in plastic bags in sealed 55-gal. drums.

Samples were taken at approximately 3-month intervals for 3 years, making a total of 12 test periods. Each wheat sampling was milled into four fractions: 90% extraction patent flour, low-grade flour, shorts, and bran. For the tocopherol analyses, representative samples of the whole grain and of each milling fraction were canned under nitrogen and stored at 32°F. Home-type bread and rolls were made with the patent flour to evaluate changes in baking quality (2) and nutrient content. Representative samples of bread, bread dough, rolls, and roll dough were freeze-dried, canned under nitrogen, and stored at 32°F.

Prior to extraction of the tocopherol-bearing lipid, the bran, shorts, and wheat

grain were ground for 1.5 min. in a water-cooled micromill (Chemical Rubber Company). Patent flour and low-grade flour required no further grinding. Freeze-dried bread, bread dough, rolls, and roll dough were ground in a mortar with a pestle, since the micromill reduced them to dough rather than to a fine powder. Tocopherols were extracted with absolute ethanol, partially purified by thin-layer chromatography, and the individual tocopherols determined by the gas-chromatographic separation of their trimethylsilyl ethers (4).

RESULTS AND DISCUSSION

Effects of Fumigants

The amounts of each of the four tocopherols commonly found in wheat α -tocopherol (α -T), α -tocotrienol (α -T-3), β -tocopherol (β -T), and β -tocotrienol (β -T-3) — are reported here in γ per g. of dry sample. The levels of these in wheat grain, both fumigated and that held as controls, at each of the twelve sampling periods, are shown in Fig. 1. A t-test on paired results for each treatment compared to the control showed no significant differences at the 5% level (5). This was true with both the ambient and the refrigerated controls. We conclude that fumigation in storage did not affect the tocopherol content of wheat.

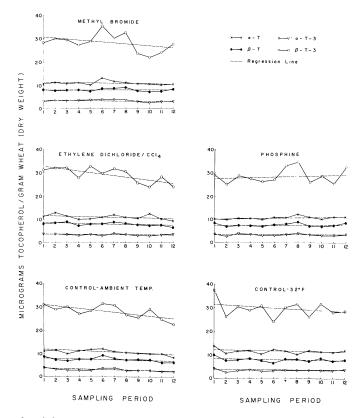


Fig. 1. Tocopherols in stored wheat grain.

Effect of Storage

Some loss of tocopherols during storage is to be expected, both as a result of their own oxidation and as a secondary effect of fatty acid oxidation. The regression line through the data for each form (Fig. 1) does in fact have a negative slope for most treatments. These negative slopes differed significantly (5% level) from zero for α -T, α -T-3, and β -T-3 in the ambient control, and for β -T and β -T-3 in the wheat treated with ethylene dichloride/CCl₄. The slopes for all forms in the phosphine-treated wheat and for α -T-3 in the refrigerated control were positive, but did not differ significantly from zero.

Effect of Milling

In view of the nonsignificant effects of fumigation and the minor effects of storage time, the tocopherol content of each milling fraction was averaged to give the data in Table I. The tocopherol forms were not equally distributed in the grain; they appeared in the wheat milling fraction in amounts reflecting a concentration of the saturated forms in the germ and the unsaturated forms in the bran and endosperm. Tocopherol recoveries from the milling process were estimated by comparing a theoretical value calculated from the milling yields (Table II) and the composition of each milling fraction (Table I) with the amount actually found in the whole grain. The calculated distribution of each wheat tocopherol in the four milling fractions, along with the estimated recoveries, is given in Table II. These calculated recoveries ranged from 87.3 to 109.1%.

TABLE I. TOCOPHEROL CONTENTS OF WHEAT GRAIN AND MILLING FRACTIONS^a (dry-weight basis, N=60)

Item	α-Τ	α-Τ-3	β-Τ	β-Τ-3
Wheat grain	11.0±0.97	3.3±0.43	7.8±0.76	28.7±3.14
Bran	17.4±4.06	10.5±2.17	12.7 ±1.47	64.0±10.06
Shorts	47.2±10.43	8.1±1.67	35.4±5.55	48.1±7.44
Low-grade flour	12.0±2.64	2.2±0.55	9.3±1.36	24.3±3.71
Patent flour	2.8±1.12	1.4±0.69	2.0±0.83	21.8±3.47

 $^{^{}a}\gamma$ per g. $^{\pm}$ S.D.

TABLE II. DISTRIBUTION OF WHEAT-GRAIN TOCOPHEROLS AMONG MILLING FRACTIONS^a

Milling Fraction	Milling Yield %	α-T ^b	α-T-3 ^b	β-T ^b	β-T-3 ^b
Bran	17.1	3.0±0.70	1.8±0.38	2.2±1.10	10.9±1.78
Shorts	8.0	3.8±1.50	0.65±0.18	2.8±0.49	3.8±0.89
Low-grade flour	7.6	0.91±0.10	0.17±0.04	0.71 ±0.10	1.8 ±0.28
Patent flour	67.1	1.9±0.76	0.94±0.27	1.3±0.32	14.6 ±2.50
Total		9.6	3.5	7.0	31.1
Recovery, %		87.3	109.1	89.7	108.4

^aγ in milling fraction per g. of dry wheat grain.

 b_{γ} per g. wheat ± S.D.

TABLE III.	EFFECT OF BAKING ON TOCOPHEROL CONTENTS			
OF BREAD AND ROLLS ^a				
	(dry-weight basis N=25)			

Sample	α-Τ	α-Τ-3	β-Τ-3	γ -T ^b	δ-Τ
Bread dough	4.0±1.48	1.3±0.31	13.2±2.28	11.2 ±1.84	4.4±1.12
Bread	3.6±1.16	1.5±0.32	10.8±1.42	12.8±2.65	5.3±1.13
Roll dough	10.3±1.61	2.9±0.29	13.2±1.86	42.3 ±6.24	16.4±2.38
Rolls	10.8±1.59	2.9±0.64	11.9±1.17	49.2 ±5.76	16.9±2.57

 $^{^{}a}\gamma$ per g. \pm S.D.

Effect of Baking

The baking process may be expected to destroy tocopherols. Moore et al. (6), for example, found a decrease of 36% in the total tocopherols of bread compared to that of wheat flour (α -T, 51% loss; α -T-3, 44%; β -T, 38%; and β -T-3, 29%). Menger (7) reported a 5% decrease in the total tocopherol from the baking of white bread. In the present work, bread and rolls prepared from patent flour from all five treatments sampled in the last five sampling periods were analyzed for tocopherols before and after baking (Table III). The amounts of tocopherols found indicated no losses except for β -T-3 (bread, 18.2%; rolls, 9.9%). These samples contained, in addition to the forms derived from wheat, substantial amounts of α -T, gamma tocopherol (γ -T), and delta tocopherol (δ -T) contributed by the shortening, which was present in bread dough at 2% per 100 g. of flour, and in roll dough at 8%. The increases seen in the forms derived from the shortening may have come from the small amounts of fat used to grease the pans prior to baking.

Acknowledgments

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b Includes a small amount of β -T.