effects was much less dramatic than those resulting from bisulfite treatment.

Sensory Evaluation. The cookies and muffins made with untreated flour had normal flavors. However those made with bisulfite-treated flour were perceived as having slight off-flavors (fruity and watermelon rind) by two panelists; the other two panelists did not detect any off-flavors.

Conclusions. This study demonstrated that it is possible to reduce the level of DON in contaminated wheat by chemical and physical treatment. Considerable reductions in DON levels were achieved by tempering contaminated wheat with sodium bisulfite (equivalent to ca. 540 μg/g added) prior to milling, although rheological and baking properties indicated that the resulting flour for this treatment level would be unsuitable for general commercial use. Milder treatment conditions for sodium bisulfite or some of the other treatments may have the potential for affording flour of normal commercial utility while still offering sufficient reduction in DON to below the current guideline limit (Canadian) of 2 μg/g in uncleaned soft wheat, should the natural level be above this value. These treatments also show promise for use in decontaminating grains destined for animal feeds.

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Registry No. DON, 51481-10-8; $NaHSO_3$, 7631-90-5; SO_2, 7446-09-5; HCl, 7647-01-0; NH_4OH, 1336-21-6; H_2O_2, 7722-84-1; NaOCl, 7681-52-9; ascorbic acid, 50-81-7.

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Reduction in Levels of Deoxynivalenol in Contaminated Corn by Chemical and Physical Treatment

J. Christopher Young

Field corn artificially inoculated with the fungus *Fusarium graminearum* was treated by various chemical and physical means. Moist ozone (1.1 mol %) in air effected a 90% reduction in deoxynivalenol (DON, vomitoxin) levels, at ca. 1000 μg/g, after 1 h, while dry ozone effected only a 70% reduction after the same treatment time. Total destruction was achieved after 0.5 h with 30% chlorine (v/v in nitrogen). Prolonged (18 h) exposure to ammonia was required to obtain an 85% reduction in DON levels. Thermal treatment, by microwave or convection, achieved 50–90% reductions. The inclusion of ammonium carbonate with heat treatment afforded only slight improvements in destruction of DON. Nearly complete reduction could be achieved by addition of aqueous sodium bisulfite with the extent dependent upon concentration and contact time.

The trichothecene mycotoxin 4-deoxynivalenol (DON, vomitoxin, 3α,7α,15-trihydroxy-12,13-epoxytrichothec-9-en-8-one) is a naturally occurring metabolite produced by the fungus *Fusarium graminearum* Schwabe on a variety of cereal grains and is known to be associated with several diseases in animals and is related to other trichothecenes that affect humans as well (Ueno, 1983).

Although chemical and physical treatments of grains contaminated with other mycotoxins such as aflatoxin (Doyle et al., 1982) have been investigated in some detail, there have been few reports on such treatments where DON has been present. Bleaching of flour with chlorine in a commercial mill afforded only a modest 10% reduction in DON concentration (Young et al., 1984). In a preliminary study, Swanson et al. (1984) reported some lowering

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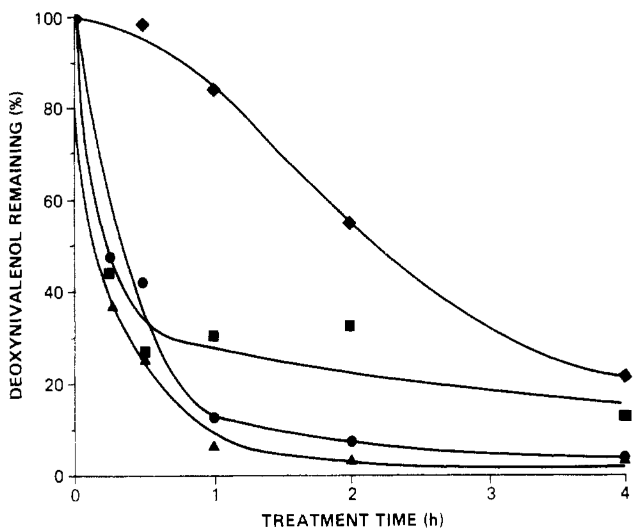

Figure 1. Reduction in deoxynivalenol content of contaminated corn (ca. 1000 μg/g) by treatment with ozone. Treatment conditions: (●) moist corn, moist ozone; (▲) moist corn, dry ozone; (■) dry corn, moist ozone; (♦) dry corn, dry ozone.

of DON levels in corn treated with sodium bisulfite.

Other studies on decontamination of DON-containing cereals have dealt mainly with the food-processing aspects (cf. Young et al., 1986). This paper describes the results of studies on some chemical and physical treatments of DON contaminated corn.

MATERIALS AND METHODS

Corn. Several unhusked ears of corn that had been artificially inoculated with *F. graminearum* (Neish et al., 1983) were harvested, ground up, and stored frozen (−18 °C) until treated or analyzed. Such samples are hereafter referred to as "moist". Some material was dried overnight in a vacuum oven (50 °C) prior to treatment.

Analysis for Deoxynivalenol. Corn samples were extracted and analyzed by the high-performance liquid chromatographic method of Neish et al. (1983). Estimations of DON were made by comparison of peak heights from injected samples with those of standards.

Treatment of Contaminated Corn. Ozone (1.1 mol %) was generated in dry air and passed through 2-g corn samples (moist or dry) at 150 mL/min. In some instances, the ozone was moistened after generation by being passed through water with a sintered-glass bubbler apparatus. A 250-mL round-bottom flask containing 2 g of moist sample was evacuated under vacuum, filled with chlorine or ammonia (100% or diluted with nitrogen), reevacuated, and filled to ambient pressure and temperature. After being allowed to stand in the dark at room temperature for the treatment period, the sealed flask was evacuated and flushed well with air. Some treatments were conducted on 2-g samples in sealed glass tubes in an oven. Other samples were mixed with ammonium carbonate (10% added by weight) prior to heat treatment. Microwave treatments were conducted at 2450 Hz on 2-g moist samples in open glass tubes; the power was turned off when the sample reached the desired temperature. Varying amounts of sodium bisulfite were dissolved in 6 mL of water and added dropwise and with extensive stirring to 10-g samples of corn contaminated at ca. 500 μg/g; the samples were then kept at room temperature for 6 days. In another experiment, samples were taken daily for up to 1 week.

RESULTS AND DISCUSSION

The effects of ozone on levels of DON in corn contaminated at ca. 1000 μg/g are summarized in Figure 1. The presence of moisture is critical for degradation of DON; although relatively little reaction between ozone and DON was apparent if both the gas stream and sample were dry, when either water-saturated ozone or regular sample (ca. 50% moisture) was used, significant degradation was observed. In another study, Young et al. (1986) observed that moist ozone had little effect on the DON content of contaminated wheat because of low moisture content (ca. 12% in the sample) and large sample size (1 kg) in comparison with the amount of ozone available. Chlorine was also able to effect a reduction in DON levels but only at gas concentrations of greater than about 1% (Figure 2). The lack of significant reduction in DON levels in contaminated wheat under normal commercial milling and bleaching conditions (Young et al., 1984) was likely due to the low levels of chlorine (0.1–0.5%) and relatively short contact times (several minutes) used. Ammonia (100% at room temperature) effected reductions in DON levels but only after lengthy exposure (9 and 85% after 1 and 18 h, respectively).

Prompted by the observations that the cookies containing ammonium carbonate in the dough showed the greatest reduction in DON levels upon baking (Young et al., 1984) and that ammonia also caused DON to disappear (vide supra), the combined effects of heating and ammonium carbonate were investigated. The data (Table I) show that the major effect was due to heat. In another

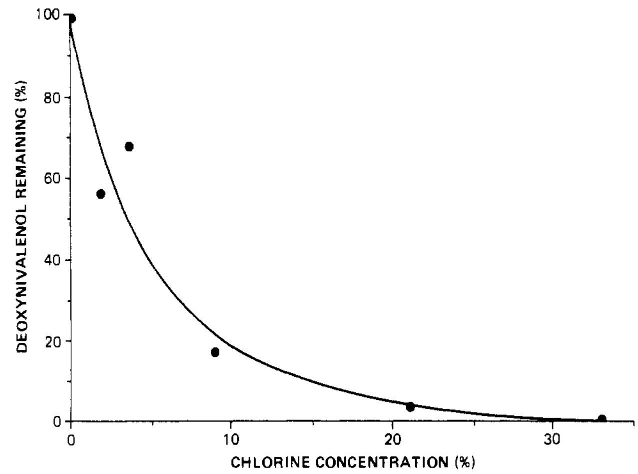

Figure 2. Reduction in deoxynivalenol content of contaminated corn (ca. 1000 μg/g) by treatment with chlorine diluted with nitrogen (v/v).

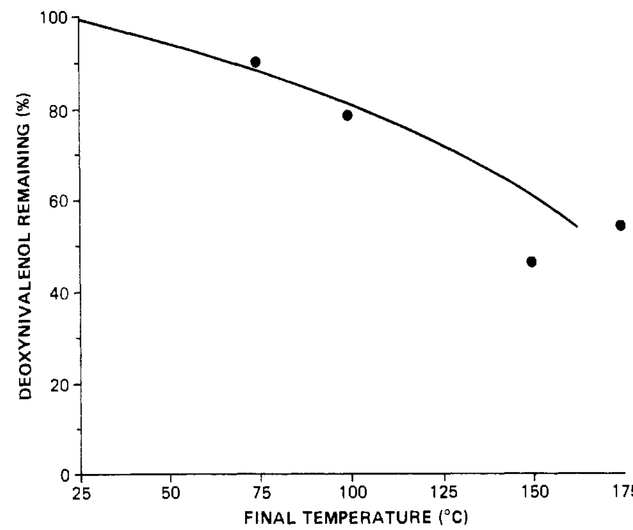

Figure 3. Reduction in deoxynivalenol content of contaminated corn (ca. 1000 μg/g) by treatment with microwaves.

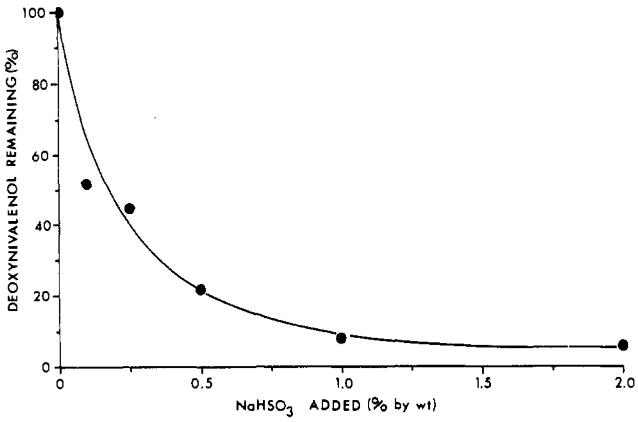

Figure 4. Reduction in deoxynivalenol content of contaminated corn (ca. 500 $\mu g/g$) after 6-day treatment of 10-g samples with sodium bisulfite in 6 mL of water. Results are averages of duplicate experiments.

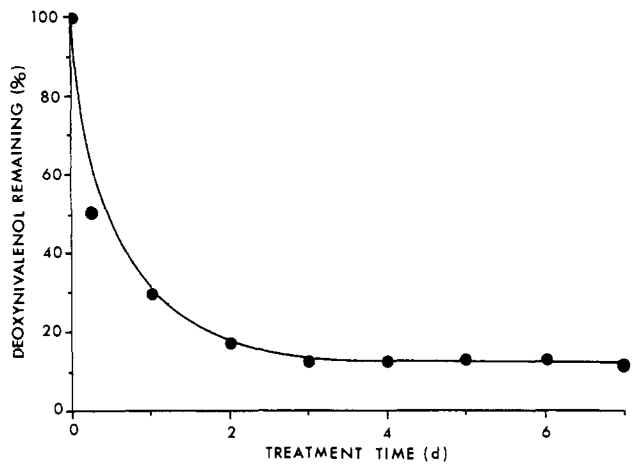

Figure 5. Reduction in deoxynivalenol content of contaminated corn (ca. 500 $\mu g/g$) by treatment of 10-g samples with 100 mg of sodium bisulfite in 6 mL of water. Results are averages of duplicate experiments.

study, Friend et al. (1984) observed that steam pelleting (at 90 °C) of DON-contaminated wheat in the presence of ammonium carbonate (1% by weight) did not alter the toxic effects of the resultant feed on pigs. Thus, under the conditions used in these various studies, the amount of ammonia released was insufficient to account for any destruction observed.

Microwave treatment was partially successful in lowering DON levels (Figure 3), with greatest effect occurring at the highest temperatures. The greater effect of convection oven applied heat compared with microwave heating can be attributed to contact time; once the final temperature was achieved under microwave conditions (several minutes), the sample was allowed to cool, whereas in the convection oven, the sample remained at the treatment temperature for 1 h.

Table I. Relative Amounts of Deoxynivalenol Remaining in Contaminated Corn[a] after Treatment with Heat and Ammonium Carbonate

	% deoxynivalenol remaining[b]	
temp, °C	heat	heat and ammonium carbonate[c]
70	87	88
100	5	14
132	19	8

[a] Field corn contaminated at ca. 1000 $\mu g/g$. [b] After 1 h relative to untreated contaminated corn analyzed simultaneously. [c] 10% Ammonium carbonate added to corn.

The effect of sodium bisulfite in substantially lowering DON levels in highly contaminated corn is in agreement with those observed in corn by Swanson et al. (1984) and in wheat by Young et al. (1986). After 6 days of treatment, nearly complete reduction was achieved by the addition of 1% or more (w/w) of sodium bisulfite (Figure 4). The disappearance of DON went smoothly (Figure 5) with time until a steady-state value was achieved after 3 days.

This study showed that a variety of chemical and physical treatments can effect a reduction in levels of DON in contaminated corn. The treatments may be too drastic for grains destined for human consumption but could find utility in animal feed production.

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Registry No. DON, 51481-10-8; O_3, 10028-15-6; Cl_2, 7782-50-5; $(NH_4)_2CO_3$, 506-87-6; $NaHSO_3$, 7631-90-5.

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