CONTROL OF PESTS AND QUALITY ASPECTS IN COCOA BEANS AND HAZELNUTS AND DIFFUSION EXPERIMENTS IN COMPRESSED TOBACCO WITH CARBON DIOXIDE UNDER HIGH PRESSURE

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ABSTRACT

In the food processing industry, such raw agricultural products as cocoa beans, hazel nuts and tobacco require quick disinfestation prior to storage. The disinfestation method must not cause any decrease in quality or any build-up of chemical residues. Exposure to carbon dioxide (CO₂) under pressure of 20–40 bar for a few hours is a recently developed and effective control method for this purpose.

Experiments with caged pest insects (developmental stages and adults) of 12 species (Lasioderma serricorne, Oryzaephilus surinamensis, O. mercator, Tribolium castaneum, T. confusum, Cryptolestes turcicus, Trogoderma granarium, Corcyra cephalonica, Ephestia elutella, E. cautella, Plodia interpunctella and Sitotroga cerealella) were carried out on 1 t of bagged product in a 3-m³ chamber. At about 10°C under 20 bar of CO₂ the lethal treatment period was slightly longer (3 h) than at 20°C. At 20°C and 30 and 37 bar, complete control was achieved within 1 h and within 20 min, respectively.

The components and possible alterations of aroma in cocoa beans and hazelnuts were tested, as were resistance to deterioration, triglyceride composition and crystallisation behaviour. The quality of the cocoa beans had not changed following these treatments. Similar results occurred with hazelnuts, excepting the tendency of treated ones to turn rancid earlier than untreated ones. In the centre of compressed tobacco, a slight delay in even distribution of the gas was observed.

INTRODUCTION

Both growing public awareness of insecticide residues in food and discussion of a ban on the use of methyl bromide encourage the search for alternative methods of pest control. The necessity of finding ways to protect stored products is especially acute for such high value commodities as confectionary and sweets, both quantitatively and qualitatively. In addition to actual economic loss, another critical factor in the food processing industry is possible damage to the reputation of the manufacturer.

To address this problem, Stahl *et al.* (1985) and Stahl and Rau (1985) described a new process, using carbon dioxide (CO₂) under high pressure, for residue-free pest control. According to follow-up studies, the quality of the treated products was not affected detrimentally (Gerard *et al.*, 1988a, b, 1990). Furthermore, using CO₂ under high pressure has the added practical advantage of requiring extremely short lethal exposure times, ranging from minutes to only few hours (Reichmuth and Wohlgemuth, 1994; Prozell and Reichmuth, 1990, 1991).

MATERIALS AND METHODS

High pressure chamber

All experiments were conducted in a 3-m^3 high-pressure chamber (CARVEX) connected to a tank of liquid CO_2 placed on a balance (Fig. 1).

Prior to its injection into the chamber, the CO₂ was warmed in the regulator. Pressure regimes for the different experiments were adjusted as required (Table 1), and at the end of each exposure period the pressure was released within about 8 min.

The exposure time included the time needed to obtain the final pressure, the time held at constant pressure and the time needed to release the CO_2 (Fig. 2). The temperature in the chamber was recorded during exposure.

Insects

Experiments were undertaken using all developmental stages of a mixture of 12 insect species, all important pests in the confectionary and tobacco industries (Table 1).

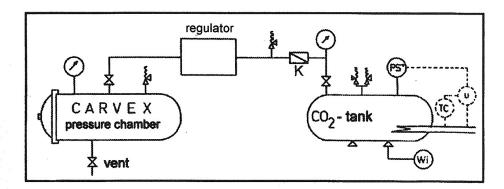


Fig. 1. Pressure chamber of about 3 m³ designed to hold CO_2 at 20 and 37 bar, liquid CO_2 supply tank and regulator unit (Wi = balance, PS+, u, TC = instrumentation to adjust temperature and pressure in the tank, K = valve (after Gerard *et al.*, 1990).

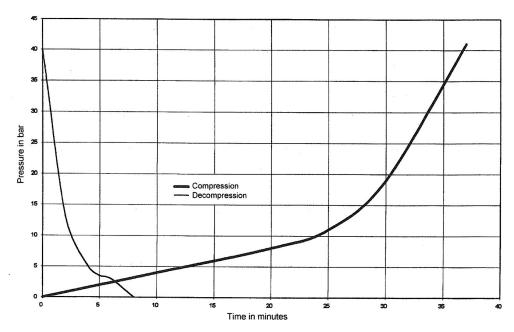


Fig. 2. Time required for compression and decompression in experiments at 37 bar; in other experiments, when the pressure was lower, the corresponding times for pressure build-up and decrease were shorter.

Adult insects of each of the following species: O. mercator, C. turcicus, T. granarium, C. cephalonica, E. elutella, E. cautella, S. cerealella and P. interpunctella were exposed together with the developmental stages. They were introduced in tubular, stainless-steel wire-mesh cages (length 10 cm, diameter 1 cm) closed with stoppers.

For the beetles *Oryzaephilus surinamensis*, *Tribolium castaneum*, *T. confusum* and *Lasioderma serricorne*, trials were carried out using eggs, young larvae, larvae, pupae and adults in separate cages.

At the beginning of each trial the chamber was loaded with cocoa beans and the cages were then placed among the cocoa beans in three areas: the front, middle and back of the chamber. The chamber was then closed and pressurized with CO_2 . Tests were carried out at 10 and $20^{\circ}C$.

Following treatment, the pressure was released and the cages removed. The samples were then held at 26°C and 75% r.h. For the following 14 weeks, they were observed weekly for survivors.

Quality control of cocoa beans and hazelnuts

Several trials were performed to determine any possible changes in the quality of treated cocoa beans and hazelnuts. At all experimental pressures and exposure times, the composition and changes in several quality parameters were tested.

Aroma analysis was done by gas chromatography (Ziegleder, 1991). Organoleptic assessment of raw and roasted cocoa beans was carried out by a team of six experienced

persons and using chemical analysis. The aroma of hazelnuts was analysed by gas chromatography. Amadori compounds in raw cocoa (the preliminary stages of aroma) were analysed by gas chromatography (Ziegleder and Oberparleiter, 1996). The storability of hazelnuts was evaluated using an accelerated storability test at 35°C for 3 months. The induction time of extracted hazelnut oil was evaluated by means of the ranzimat test. Hexanal in hazelnuts was determined by headspace-gas chromatography. The gradient of cocoa butter, extracted from cocoa beans, was determined by the isotherme dsc method (Ziegleder, 1990). HPLC-trygliceride determination in oil extracted from both treated and untreated nuts was performed (Ziegleder *et al.*, 1996).

The rate of distribution of carbon dioxide within the commodity

A 1-m³ tobacco bale $(0.8 \times 0.6 \times 0.5 \text{ m})$ containing a stainless steel tube (3 mm diameter and 1 mm centre bore) (Ulrichs *et al.*, 1997b) was treated with CO₂ at 20 bar. The results of the diffusion measurements into the centre of the bale were recorded.

RESULTS

Insect mortality

All insects in the untreated control samples showed normal development. The results of CO₂ treatment of the insects under different pressures are presented in Fig. 3 and Table 1.

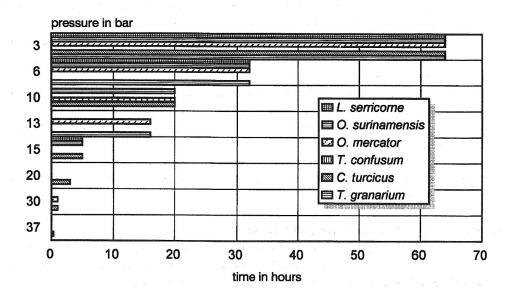


Fig. 3. CO_2 treatments at different pressures and exposure times, lethal to all stages of six stored-product beetles at 20°C.

TABLE 1 Results of $\rm CO_2$ treatments for different exposure times at 10°C and 20°C of all developmental stages of 12 stored-product insects in a 3-m³ pressure chamber

| Species | Tem- | CO ₂ pressure in bar (Exposure time in h) | | | | | | | | |
|-------------------|------------------|--|--------|----------|---------|--------|--------|--------|-------|-----------|
| | perature (°C) | 3 (64) | 6 (32) | 10 (20) | 13 (16) | 15 (5) | 20 (8) | 20 (3) | 30(1) | 37 (0.33) |
| L. serricorne | 10 | _ | _< | X | _ | х | _ | х | 0 | 0 |
| | 20 | X | X | | 0 | _ | 0 | 0 | 0 | 0 |
| O. surinamensis | 10 | - | _ | X | _ | X | _ | Х | 0 | 0 |
| | 20 | X | X | _ | 0 | - | 0 | 0 | 0 | 0 |
| O. mercator | 10 | | - | _ | _ | | _ | - | - | |
| | 20 | X | X | _ | X | _ | 0 | 0 | X | 0 |
| T. castaneum | 10 | - | _ | _ | _ | _ | _ | _ | _ | _ |
| | 20 | 0 | 0 | - | 0 | _ | 0 | 0 | 0 | 0 |
| T. confusum | 10 | _ | _ | X | - | 0 | | 0 | 0 | 0 |
| | 20 | 0 | 0 | _ | 0 | - | 0 | 0 | 0 | 0 |
| C. turcicus | 10 | | _ | x | _ | х | | х | X | х |
| | 20 | X | 0 | _ | 0 | _ | 0 | X | X | X |
| T. granarium | 10 | _ | _ | - | _ | | _ | _ | _ | _ |
| | 20 | X | x | - | X | _ | 0 | 0 | 0 | 0 |
| C. cephalonica | 10 | _ | _ | 0 | - | 0 | _ | 0 | 0 | 0 |
| | 20 | - | - | _ | - | - | - | 0 | 0 | 0 |
| E. elutella | 10 | _ | _ | 0 | | 0 | _ | 0 | 0 | 0 |
| | 20 | 0 | 0 | _ | 0 | _ | 0 | 0 | 0 | 0 |
| E. cautella | 10 | _ | _ | 0 | _ | 0 | | 0 | 0 | 0 |
| | 20 | 0 | 0 | - | 0 | _ | 0 | 0 | 0 | 0 |
| S. cerealella | 10 | _ | _ | _ | _ | _ | _ | | _ | _ |
| | 20 | | | - | - | _ | - | Ö | x | 0 |
| P. interpunctella | 10 | _ , | | 0 | _ | 0 | _ | 0 | 0 | 0 |
| | 20 | 0 | 0 | _ | 0 | | 0 | 0 | 0 | 0 |

x = survivors; 0 = no survivors; -= no experiment.

Treatments at 20°C

High pressure treatment at 37, 30 and 20 bar was carried out at 20°C. Treatments of 20 min at 37 bar, 1 h at 30 bar and 3 h at 20 bar all produced 100% mortality of all test insects except *Cryptolestes turcicus*. Also, at 30 bar and 1-h exposure *O. mercator* was not completely controlled.

High pressure treatment was carried out at 20°C for 8, 16, 32 and 64 h. After 16, 32 and 64 h at 13, 6 and 3 bar, respectively, complete mortality in all tests was achieved for the moths (*Corcyra cephalonica, Ephestia elutella, E. cautella* and *Plodia interpunctella*) and

the two *Tribolium* species (*T. castaneum* and *T. confusum*). At 8 h and 20 bar exposure, no survivors of the other beetle species were found.

C. turcicus survived only the 64-h treatment at 3 bar; O. surinamensis and L. serricorne were controlled at 16 h and 13 bar; and Trogoderma granarium and O. mercator survived 16 h at 13 bar. Despite delays in the hatching of the eggs, it was concluded that some eggs were not controlled.

Treatments at 10°C

High pressure treatment at 37, 30, 20, 15 and 10 bar was carried out at 10°C. At 37, 30, 20, 15 and 10 bar, and at 20 min, 1, 3, 5 and 20 h, respectively, 100% mortality of the moths was obtained. These pressures and exposures times were insufficient to produce 100% mortality of the test beetles.

L. serricorne and O. surinamensis failed to survive 30 and 37 bar at 1 h and 20 min, respectively, and survivors of T. confusum were found after treatment at 10 bar at 20 h. Only C. turcicus survived all of the tested pressures and exposure times.

Quality control

The quality criteria used for cocoa beans did not reveal any significant changes following the treatments. The treated hazelnuts had a tendency to turn rancid earlier than did the untreated ones. All the other quality criteria of hazelnuts also remained unchanged.

Rate of distribution of carbon dioxide

It was shown that the build-up of high CO_2 concentration in the centre of the tobacco bale did not correspond directly to the rapid increase in CO_2 concentration around the bale within the chamber. Shortly after starting pressurisation, an area of compressed air into which CO_2 did not penetrate remained in the centre of the tobacco bale. After 2.5 h the CO_2 content increased in this area as well.

DISCUSSION AND CONCLUSIONS

Ferguson and Hawkins (1949), Johnson and Quastel (1953) and Carpenter (1954) were the first to describe the toxic action of inert gases under increased pressure. They mentioned narcotic effects after treatment with these gases; presumably, then, the death of treated insects during treatment under high pressure is due to prolonged and intense narcosis. In addition, the destruction of cell membranes during decompression causes severe damage. The mortality results of the experiments presented here are similar to those reported by Prozell and Reichmuth (1990, 1991) on *S. granarius*.

Post-treatment quality analyses of cocoa beans and hazelnuts showed that treated hazelnuts have a tendency to turn rancid earlier than untreated ones; cocoa beans showed no quality change at all.

It is difficult to implement in practice the laboratory findings with CO₂ and high pressure because the laboratory results were achieved in small chambers of 150 ml, where

there was a short time for build-up of pressure and decompression and the temperatures were fixed. Findings by Ulrichs (1994) and Ulrichs *et al.* (1997a) indicated the importance of the decompression speed on mortality, and this was also mentioned by Nakakita and Kawashima (1994).

The findings on the rate of distribution of the CO₂ presented here indicate that it depends on the nature and mass of the treated product. When the air was initially compressed, the centre contained low CO₂ concentrations insufficient to control the insect pests (Prozell and Reichmuth, 1991). Later the CO₂ concentration increased, and four phases of penetration which follow the classical transport phenomena (Bird *et al.*, 1960) can be described: the filling of the substrate pores, followed by the balancing of the pressure; the diffusion of CO₂ into the interstitial spaces, balancing out differences in gas content; the diffusion of oxygen out of the pores; and lastly even mixing by means of diffusion.

With compressed products or commodities with small interstitial air spaces, the air within the product is quickly compressed as CO₂ is introduced into the chamber. This is due to the pronounced pressure gradient from the outside of the product to the inside. This compression occurs so quickly that the interstitial air cannot diffuse out from the centre during the short time following the initiation of treatment. After even pressurisation diffusion processes slowly lead to gas exchange within the product. This exchange time must be added to the actual treatment time in carrying out effective pest control.

The time required to obtain the necessary CO₂ concentration for controlling the insects may be longer for compressed products because more time will be required for the first three phases (Fig. 4). Prior to high pressure treatment, it is necessary to identify the pest and the developmental stages present in the commodity. The treatment time must be adjusted accordingly to obtain the necessary lethal exposure.

Content of carbon dioxide in vol.-%

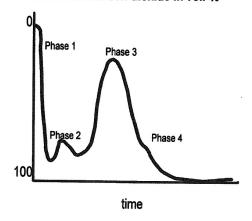


Fig. 4. Change in CO₂ concentration at the centre of a bale of compressed tobacco in a fumigation chamber during treatment with high pressure at 20°C (Modelled after Bird *et al.*, 1960).

In conclusion, unlike treatment with other classical insecticides and toxic fumigants, this treatment can be used as a preventive method to ensure pest-free cocoa beans and other foods without leaving chemical residues. In addition, this method has a low probability of causing change in quality, provided that the moisture content of the commodity is low.

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