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CRITICAL LIMITS OF SEALING FOR SUCCESSFUL APPLICATION OF CONTROLLED ATMOSPHERE OR FUMIGATION

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ABSTRACT

A fundamental requirement for the successful application of gaseous treatments to control stored-product insects is a well-sealed structure. A flexible fumigation structure of 7.5 m^3 capacity marketed as the "GrainPro Cocoon™" that is used for outdoor storage of stacked commodities was used to demonstrate the critical limits of the degree of sealing using the variable pressure test. Time in minutes for the half-life pressure decay was correlated with daily ventilation rates of O_2, CO_2, PH_3 and MB. The ventilation rates were tested using different sizes of cross-section leak areas with orifices of 1.6 mm, 3.2 mm and 6.4 mm i.d. To evaluate the influence of temperature on the rate of gas exchange, the cube was tested when it was under cover by providing shading to minimize the direct solar heating effect, and when it was exposed to direct solar heating. Oxygen infiltration rate for modified atmospheres was 0.5% O_2 day^{-1} at 5 min half-life pressure decay when the chamber was under shade. For the same level of gastightness, gas loss was 0.8% O_2 day^{-1} when exposed. CO_2 loss rate was 2% day^{-1} at 5 min half-life pressure decay when the cube was under shade and 3% day^{-1} when the cube was exposed. Loss rate of PH_3 from the cube for the shaded conditions was 100 ppm day^{-1} at a 3-min half-life pressure decay time. A sealing equivalent to 4.5 min half-life pressure decay time was required when exposed. The MB loss rate of 1.0 g m^{-3} day^{-1} was equivalent to ~ 5 min half-life pressure decay time. At the time tests were carried out with MB, ambient temperature differences were not large enough to show different loss rates between the shaded and the exposed cube.

INTRODUCTION

A fundamental requirement for the successful application of modified atmosphere (MA), controlled atmosphere (CA), and fumigation treatments to control stored-product insects is a well-sealed structure, where the desired gas concentration should be maintained at a sufficient level. The requirement for gastight storages for application of CAs and MAs appears to be more critical than the requirement for application of fumigants (Bond, 1984). In the sealed storage also known as hermetic storage, the structure should be sufficiently airtight to enable insects and other aerobic organisms in the grain-mass to reduce oxygen (O_2) concentrations below those permitting insect development.

Fumigants have been used for many years with limited requirements for structural tightness. The consequences of poorly sealed storages under fumigation should be considered in view of the development of insect resistance to fumigants in poorly sealed structures (Banks and Desmarchelier, 1979; Banks, 1981; Zettler, 1993).

Methods to determine gastightness have been investigated to analyze the energy requirements of buildings. The dynamic characteristics of air infiltration into buildings have been studied in order to predict the heating and cooling load of seasonal energy requirements (Hill and Kusuda, 1975). The ASHRAE Handbook of Fundamentals describes the air change method and the crack method in predicting air infiltration rates (Anon. 1972). The crack method or the constant pressure test is usually regarded as more accurate as long as the leak characteristics can be evaluated properly (Hill and Kusuda, 1975).

Meiering (1982) investigated a constant pressure test and a variable pressure test for measuring specific silo permeability in silage systems, where the sealed shell is designed to limit entry of O_2 to minimize losses in silage quality. Sharp (1982), using the constant pressure test, measured the gastight level of sealed structures. These tests are designed to estimate the permissible limits for effectively maintaining the gas composition in the stores during the treatment.

Gas interchange within freight containers and factors leading to gas interchange between containers and external atmosphere were detailed by Banks *et al.* (1975). They found that the relationship between applied pressure and gas leak rate gave a useful measure of gastightness. Banks and Annis (1977) developed a practical guide for storage of dry grain under MA and specified requirements for silo gastightness. Their specifications correspond to pressure decay times needed to maintain the atmospheric composition in the silos.

Prior to the gaseous treatment a decision should be made as to the degree of gastightness that is satisfactory or the gas concentrations that can be maintained under given environmental and structural conditions This decision should be weighed against the investment involved in sealing a leaky structure to prevent excessive loss of the treatment gas.

The objective of this study was to demonstrate the critical limits of degree of sealing using the variable pressure test for successful CA and fumigation treatments to control storage insects.

MATERIALS AND METHODS

Structure for gaseous treatments

A fumigatable flexible structure using a heavy-duty liner for outdoor storage of stacked commodities of 7.5 m^3 volume termed also as "GrainPro Cocoon" or "Volcani Cube" was used. Such structures commonly called Cubes, were originally designed for hermetic storage, but have also been modified for use as fumigation chambers or for modified atmosphere storage of commodities (Donahaye *et al.,* 1991). The cube was equipped with two ports; one for gas supply furnished with a 3.8 cm diameter ball valve, and the other an exhaust port.

To maintain the dimensions, the cube was supported from inside using empty cardboard, polystyrene and two standard wood boxes. The cube was erected at the Horticultural Crops Research Laboratory, (HCRL), Fresno, CA.

To evaluate the influence of temperature on the rate of gas exchange the cube was tested when it was under cover by providing shading, to minimize direct solar heating effect, and also when it was exposed to direct solar heating.

Variable pressure test

In a variable pressure test, the structure is pressurized to a value above or below atmospheric, using a fan. The air supply is then shut off and the pressure is allowed to fall by natural leakage to a new value. The time taken to fall from the high (positive or negative) pressure serves as a measure of the degree of sealing. Time elapse to half the pressure, known as half-life pressure decay-time is usually considered for comparisons of gastightness level. The cube was tested under negative pressure except for one control in positive pressure.

A vacuum cleaner capable of delivering 3 m^3 min^{-1} of air at 2,000 Pa and a "Dwyer Instruments" inclined manometer, measuring up to 1,500 Pa were used for pressure measurements.

A negative pressure was created by evacuation until the sheeting adhered to the packed material. Tests were carried out under as stable temperature and barometric conditions as possible.

Time in minutes taken for the pressure to fall between the two pressure limits was recorded. The same limits were used each time a particular leak was tested. Half life pressure decay times were correlated with daily ventilation rates of O_2, CO_2, phosphine (PH_3) and methyl bromide (MB).

Ambient conditions

Ambient temperatures were recorded with a "Datapod DP-220" data logger. Barometric pressure was recorded using a mechanical Precision Microbarograph, with weekly recording charts. The barograph was in full accordance with official local barometric recordings.

Predetermined orifices

The relationship between ventilation rate and gas exchange was studied in a series of tests with different cross-section leaks having openings of 1.6 mm, 3.2 mm and 6.4 mm i.d. orifices with wall thickness of 4 mm Removable caps controlled leak openings.

Generation of the gaseous atmospheres in the cube

Gas retention in the cube was compared using O_2, CO_2, PH_3 and MB.

For the generation of a low, 5.1% O_2 atmosphere, N_2 was used from a pressurized cylinder of 2,500 psi (178 kg cm^{-2}), maintained at a discharge pressure of 15 psi. In this way a cylinder of 7.22 m^3 could be discharged into the cube in 5 h or at a rate of 24 L min^{-1}. This gas flow rate was just sufficient to avoid pressure build up during the purge phase of N_2 while the exhaust port of 3.8 cm i.d. was kept open.

For a CO_2 enriched atmosphere, CO_2 was used from a pressurized cylinder of 1,150 psi (82 kg cm^{-2}). The CO_2 supply was maintained at 15 psi. In this way a cylinder of 13.88 m^3 could be discharged into the cube in 6 h or at a rate of 38.6 L min^{-1}. This gas flow rate was just sufficient to avoid ice formation on the regulator and pressure build up during the purge phase of CO_2 while the exhaust port of 3.8 cm i.d. was kept open. An atmosphere of 76.6% CO_2 and 4% O_2 was maintained.

For generating PH_3, 225 metal phosphide pellets each weighing 0.6 g were used to obtain a calculated 1,500-ppm of PH_3. The pellets were placed in a sachet made of filter paper and attached inside the cube by inserting it through the exit port. To enhance PH_3 release it was found necessary to wet the sachet up to twice a day. It took about 97 h to reach a concentration of 1,470 ppm.

Methyl bromide was generated from a 4 lb cylinder. It took about 5 min to discharge 300 g MB.

Determination of gas concentrations

To determine O_2 concentrations an "Oxychek-2" electrolytic sensor type oxygen monitor equipped with an internal pump delivering ~ 500 mL/min gas sample flow was used for measuring O_2 levels in the cube. For CO_2, a "Servomax" infrared analyzer was employed equipped with an infrared detector and capable measuring up to 100% CO_2. Phosphine gas concentrations were measured using a PortaSens phosphine detector equipped with an electrochemical sensor. For MB, gas samples were taken using a 100 mL syringe and injected into a gas chromatograph equipped with a 1 cc gas sampling loop and Varian flame ionizing detector.

(Title? hermetic storage?)

The influence of hermetic storage on controlling insects was examined using small scale 15, 30 and 52 m^3 capacity sealed plastic structures for out-door storage of wheat, paddy and corn (Navarro *et al.,* 1995). Pressure decay rates were compared with daily CO_2 decay rates.

Presentation of results

Results were recorded as time in minutes taken for the pressure to fall between the two pressure limits (set-points?). The same pressure limits were used each time when a particular orifice size was tested. Half-life pressure decay times were correlated with daily ventilation rates of O_2, CO_2, PH_3 and MB.

RESULTS AND DISCUSSION

Comparative results using the variable pressure test

Half-life times of pressure decay in relation to orifice sizes are given in Fig. 1. A negative correlation was found between the leak size and the pressure decay time. A base-line control that would reflect complete airtight conditions could not be achieved for the cube. Several attempts made to improve the degree of sealing of the cube, resulted in only minor improvements without obtaining a line parallel to the horizontal axis of Fig. 1, where tests were carried out using negative pressure. Test for gastightness using positive pressure resulted in an almost straight line, but this should be attributed to the small volume displacement through the leaks, relative to the volume created under positive pressure. It is also probable that some leaks enhance air infiltration under positive pressure and others under negative pressure. Depending on their position and configuration along the walls of the cube, the higher the pressure exerted in the cube, the greater will be the gas leakage. By comparing the positive and negative pressure tests, it was concluded that the leaks in the cube were of dimensions, and at locations that would not be detected by conventional method of creating soap bubbles on the leaks. Therefore, it was decided to use the control line obtained using negative pressure as a base line for ventilation rate achieved before opening the predetermined orifices on the cube. Using predetermined orifices, the lines obtained with 1.6 mm, 3.2 mm and 6.4 mm i.d. orifices were proportional, suggesting that the total leak area of the sealed cube, could be equivalent to less than 0.8 mm^2.

Fig. 1 shows that the initial pressures for the tests were maintained in the range of 1,100 –1,120 Pa (4.5” water column) and the half-life was estimated at 555 Pa. By analyzing the data it was revealed that at 555 Pa the approximate half-life pressure decay times for the predetermined orifices of 6.4, 3.2, 1.6 mm i.d. and the control were 1, 2.5, 3.5 and 4.5 min, respectively. These values were used for correlating the gas loss rate from the cube as measured during the tests.

By using the high initial pressure, lower ranges of pressures such as the recommended 500 Pa or 250 Pa can also be compared, using the data in Fig. 1. The other reason for selecting the initial pressure as 4.5" water column was because the inclined manometer could read only up to 6" water column (~ 1,500 Pa). The vacuum pump that served in the tests also performed best in this range and the rigid structure formed using the empty boxes could not stand higher negative pressures without deformation.

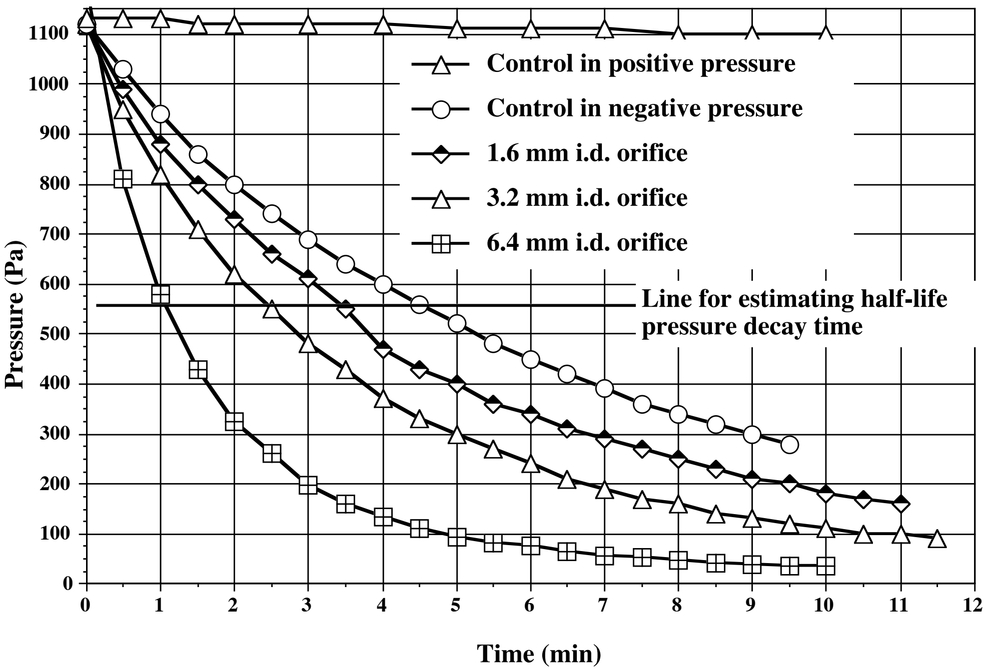

Fig. 1. Pressure decay determined for orifices of different sizes mounted on the cube. The cube was tested under negative pressure except for one control in positive pressure.

Changes in ambient temperature

Daily changes in the ambient temperature as recorded during the experiments on gas loss rate showed extremely high day temperatures of 43°C, typical for the Fresno area in July and August, with lows ranging from 18-23°C. The extreme daily difference of over 20°C in dry-bulb temperature was considered an important factor to enhance ventilation rate of the cube during the summer.

The total number of temperature changes is directly proportional to the frequency and amplitude of temperature fluctuation in the cube void space. Therefore, changes in the gas temperature were approximated by linear functions and the temperature rate of change was expressed as °C h^{-1} (Meiering, 1982). Since air infiltrates into the cube void space when a decrease in temperature occurs, the proportion of temperature rate of change with time to the initial gas temperature in the cube can be considered as the influence of temperature on the volume of air, which infiltrates into the cube.

Changes in ambient relative humidity

Daily changes in the ambient relative humidity as recorded during the experiments on gas loss showed extreme low day humidities, typical for the Fresno area, for July and August. The extreme low humidities reduced the release rate of PH_3 gas from tablets.

Changes in ambient barometric pressure

Typical daily changes in the ambient barometric pressure were recorded in inches during the experiments. Similar to changes in temperature, the increase in barometric pressure causes air infiltration into the cube. Changes in barometric pressure may be approximated by a linear function where the rate of atmospheric pressure change is expressed in Pa h^{-1} (Meiering, 1982). Since the atmospheric pressure would affect the effective total void volume of the cube, the volume of air entering the cube is proportional to atmospheric pressure change (Navarro, 1997).

In calculating ventilation rate, the basic assumption is that the volume infiltrating the cube is not restricted to leaks. Obviously in a gastight structure not equipped with a pressure relief valve, air infiltration into the cube during sudden changes in temperature or barometric pressure will be independent of the leak characteristic.

Fig. 2 expresses the barometric pressure changes in comparison to other conditions, such as maritime climate that is subject to intensive changes. Barometric pressure data were processed to express average weekly barometer rises in Pa h^{-1} and the number of weekly changes on top of each histogram. From Fig. 2 it is clear that the weekly changes in barometric pressure were very stable with about 7 changes/week in comparison with similar data collected from a maritime climate with 14-18 changes/week. Also the hourly barometric pressure variations were within the range of 1.2 to 3.1 Pa h^{-1}. On the basis of these data it is possible to estimate what the influence the barometric pressure will have on air infiltration rate or gas loss from a treated structure.

Infiltration rate of oxygen into the cube

The infiltration rate results in the exposed and shaded cubes were highly correlated to half-life pressure decay- times as they were obtained (shown?) in Fig. 1.

Membranes of concrete, plaster and plastic liners permit gas permeation and gas exchange. Variable pressure tests, as used in the present work are not capable of measuring the degree of such losses. The liner of which the cube was composed, had a permeability rate of 87 mL O_2 m^{-2} day^{-1} (Navarro *et al.*, 1994). The total surface area of the cube was 24.12 m^2 which would result in an infiltration of 2.1 L of O_2 day^{-1} or an increase of about 0.028% O_2 day^{-1}. From these calculations the main reason for the infiltration rates recorded during the experiments were probably not due to the permeation of O_2 through the plastic liner.

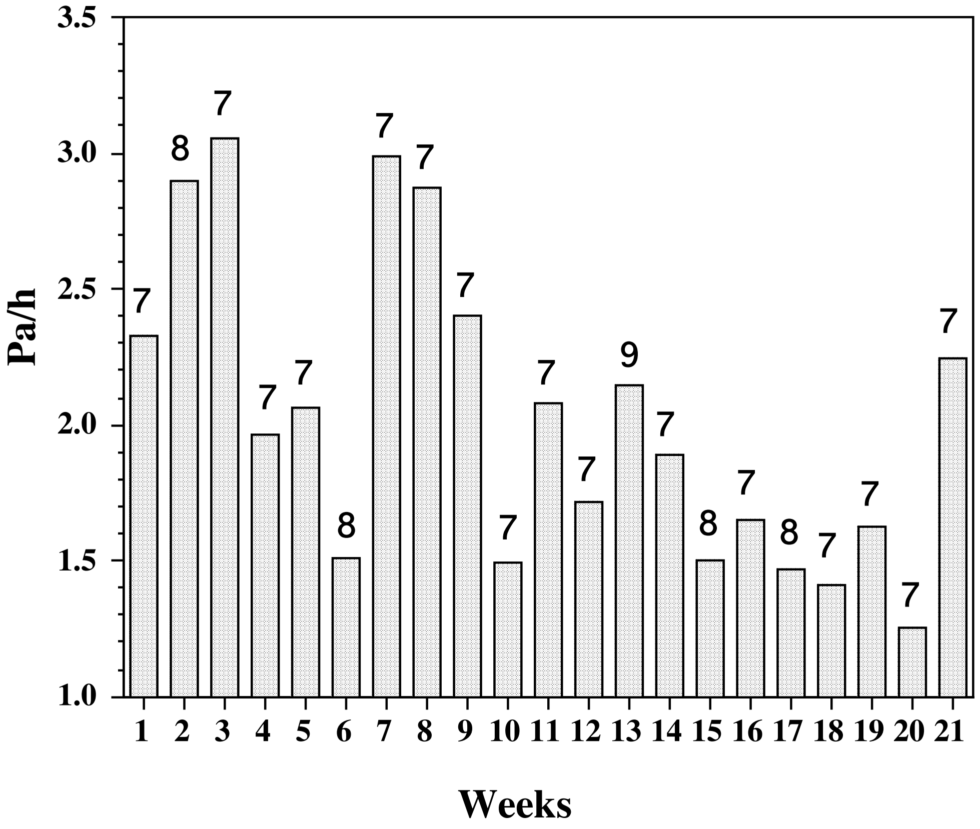

Fig. 2 Histogram showing the average weekly barometer rises expressed in Pa h^{-1} and the number of weekly changes on top of each histogram. Start of recording was on June 30, 1999 and the last reading was on November 29, 1999 (data missing on the week starting October 4).

The maximum suggested infiltration rate for MAs is 0.5% O_2/day increase (Navarro *et al.,* 1993). This level of gastightness could only be achieved for pressure test values equivalent to 5 min half-life pressure decay. For the same level of gastightness gas loss was at a higher rate when exposed to solar irradiation. The lines shown in Table 1 are at similar loss rates indicating the same degree of leakage caused by the predetermined orifices but at different intensity.

Comparisons of variable pressure tests are scarce. Navarro (1999) prepared provisional guidelines based on best estimates available in the literature. The suggested half-life pressure decay time for successful MA treatment of structures full with commodity of up to 500 m^3 was 5 min. Although data given by Navarro (1999) was calculated for larger volume structures than the cube used in the present tests, relative to the size of the cube the half-life pressure decay time should be longer than

5 min to achieve a successful MA treatment. Therefore, the 5 min half-life pressure decay time shown in Table 1 is in agreement with the suggested provisional guidelines for MA treatments given by Navarro (1999).

Loss rate of carbon dioxide from the cube

Loss rate of CO_2 from the cube is shown in Table 1. The results of gas loss rate in both exposed and shaded cubes were correlated to half-life pressure decay times as observed in Fig. 1. These tests were carried out from July 15 to August 14, 1999. For this period of time the ambient temperatures can be compared with barometric pressures in Fig. 2.

TABLE 1

Infiltration of O_2 and loss rate of CO_2, PH_3, and MB from exposed (EX) and shaded (SH) empty structure of 7.5 m^3 capacity in relation to half-life pressure decay time (min). The suggested maximum infiltration rate for modified atmospheres is 0.5% O_2 day^{-1} increase. The suggested maximum loss rate for carbon dioxide is 2% day^{-1}. PH_3 loss should not exceed 100 ppm day^{-1}. MB loss of 1.0 g m^{-3} day^{-1} was considered acceptable. For MB the shaded structure was tested in October and the exposed structure was tested in November, when ambient temperatures were lower then in October

Minutes for half-life pressure decay	Infiltration Rate (% O_2 day^{-1})		Loss Rate (% CO_2 day^{-1})		PH3Loss (ppm day^{-1})		MB Loss in (g m^{-3} day^{-1})	
	EX	SH	EX	SH	EX	SH	EX	SH
1.0	1.60	1.15	6.65	5.00	220	145	1.95	2.05
2.0	1.35	1.05	5.45	4.20	190	120	1.70	1.85
3.0	1.15	0.95	4.40	3.45	175	100	1.45	1.55
4.0	0.95	0.70	3.30	2.70	155	75	1.15	1.25
5.0	0.80	0.50	2.20	1.95	130	52	0.93	1.10

The liner of the cube had a permeability rate of 414□mL□CO_2 m^{-2}/day^{-1} (Navarro *et al.,* 1994). Since the total surface area of the cube was calculated as 24.12 m^2 the estimated loss of CO_2 was about 10 L of CO_2 day^{-1} or a decrease of about 0.133% CO_2 day^{-1}. The gas losses experienced in the present tests ranged in loss from 2 to about 7% CO_2 day^{-1}. Similar to the tests on infiltration rate of O_2 described above, it would appear that the main reason for the CO_2 losses recorded during the experiments were not due to the permeation rate of CO_2 through the plastic liner.

The data in Table 1, shows that in some orifice sections loss rates were similar, indicating the same effect of leakage caused by the predetermined orifices but at different intensity. For measurements carried out for the 2.5-min half-life pressure decay time, results in CO_2 loss rate were very similar for both the exposed and shaded cube. Examination of data on barometric pressure on the 6th week (Fig. 2) revealed a low pressure (gas exchange?) rate of 1.5 Pa h^{-1} when the exposed cube was tested suggesting that the low gas exchange could be due to the low barometric pressure rate. (what is a low pressure rate?)

For CA storage in Australia, with structures of 300 to 10,000 tonnes capacity, a decay time of 5 min for an excess pressure drop of 2,500-1,500 Pa or 1,500-750 Pa or 500-250 Pa was regarded satisfactory (Banks *et al.*, 1980). According to Banks and Annis (1980) this range of pressures was chosen as the highest usable without unduly stressing the storage fabric of the store. They commented also that above 10,000 tonnes capacity, pressure testing is difficult to carry out satisfactorily since it requires very stable atmospheric conditions. From analysis of the data presented by Banks *et al.*, (1980), it would appear that for storages with capacities in the range of 1,600-1,900 tonnes with an initial CO_2 concentration of about 60-85% for an average decay time of 11 min, the daily decay rate was ~ 4% CO_2. With a similar range of initial CO_2 concentrations in a structure of 150 m^3 capacity daily gas loss was correlated to different levels of pressure decay times (Navarro *et al.,* 1998). Their comparison resulted in a pressure decay time of 3 min for a daily decay rate of about 4% CO_2.

In the small hermetic structures tested, successful insect controls were obtained with >1% CO_2 daily decay rate which was found to be equivalent to 5 min half-life pressure decay time. Similarly comparative data was obtained using hermetic bunker storages of about 19,000 m^3 capacity, where successful results were obtained when the half life pressure decay was about 9 min (Navarro *et al.,* 1984).

For MA treatment using CO_2 a loss rate of up to 2%/day still gave acceptable insect control (Navarro *et al.,* 1998). From the provisional guidelines based on best estimates available in the literature, Navarro (1999) suggested half-life pressure decay time for successful modified atmosphere treatment of structures full with commodity of up to 500 m^3 as 5 min. The 5-min half-life pressure decay time shown in Table 1 is in agreement with the suggested provisional guidelines for modified atmosphere treatments given by Navarro (1999).

Phosphine loss rate from the cube

Loss rate of PH_3 from the cube is shown in Table 1. Prior to the test the gas concentration was increased to about 2,000 ppm. The gas loss rate results, in exposed and shaded cubes were correlated to half-life pressure decay times as they were obtained in Fig. 1. These tests were carried out from August 26 to October 13, 1999. For this period of time the ambient temperatures can be compared with barometric pressure in Fig. 2.

Based on the dosage requirement for a successful fumigation for a 10 days exposure, a loss rate of up to 100 ppm PH_3 was estimated to be within the acceptable limits for insect control. The data in Table 1 shows a similar loss rate indicating the same effect of leakage caused by the predetermined orifices but at different intensity

According to Banks and Annis (1984) daily ventilation rates tolerable in various insect control processes are estimated as 2.6% for hermetic storage, 5% for N_2 based CA, 7% for CO_2 based CA, and 10% for PH_3 fumigation. Based on the proportions of ventilation rates, this would account for ventilation rates for fumigation using PH_3 being two fold that of N_2-based CA, with the latter being as much as twice as hermetic storage.

Banks and Ripp (1984) tested sealed flat storages from 4,500 to 27,000 tonnes capacity and compared the fumigant effectiveness using PH_3 with pressure decay time from 150 to 75 Pa. Their tests resulted in successful control of insects when half-life pressure decay was 3 min for the storage capacity of 15,600 and 16,500 tonnes, whereas full insect control could not be achieved when half-life pressure decay was less than 1 min for capacities of 4,800 tonnes. From the provisional guidelines based on best estimates available in the literature, Navarro (1999) suggested a half-life pressure decay time for successful fumigation treatment of structures filled with commodity of up to 500 m^3 capacity, as 1.5 min.

For the shaded conditions a 3-min half-life pressure decay time as shown in Table 1, is equivalent to 100 ppm PH_3 loss/day, which is significantly longer than the suggested provisional guidelines for fumigant treatments given by Navarro (1999). The 1.5 min half-life pressure decay time would result in a PH_3 loss rate of about 135 ppm/day for a shaded structure. However, a better sealing equivalent to 4.5 min half-life pressure decay time would be required for the same structure, exposed to solar irradiation. Results shown in Table 1 were obtained in correlation to the different gastightness levels tested and reflect the most recent updated information on PH_3 loss. Therefore it is recommended that successful PH_3 treatments be based on a minimum loss rate of 100 ppm day^{-1} and the half-life pressure decay time for small structures adjusted to 3 min.

Methyl bromide loss rate from the cube

Loss rate of MB from the cube is shown in Table 1. Prior to the test on loss rate of MB from the cube, the gas concentration was increased to about 40 g m^{-3}. The gas loss rate results, in both situations (exposed and shaded) were correlated to half-life pressure decay times as they were obtained in Fig. 1. Tests on the shaded structure were carried out during October 14-31 and test on exposed structures during November 1-17, 1999. For this period of time the ambient temperatures can be compared with barometric pressure in Fig. 2.

Results in Table 1 show a similar loss rate indicating the same effect of leakage caused by the predetermined orifices. However their intensity was in the reverse order of the results obtained in tests with N_2, CO_2 and PH_3. In Table 1 the gas loss rate from the shaded cube was significantly higher than from the exposed cube.

Examination of temperature data during October 14-31 reveals a higher ambient temperature when the shaded cube was tested than during November 1-17 when the exposed cube was tested. This clearly shows that the expected high temperature variation could not be achieved for the exposed cube suggesting that the low gas exchange is due to the lower temperatures recorded during the same period.

With sorptive fumigants like MB, a pressure decay test for the assessment of gas retention is not a precise concept since besides the gas loss through leaks, sorption by the treated material is a major cause for drop in gas concentration. Wheat flour treated with MB in experimental jars, resulted in sorption of 70% of the initial gas concentration after 24 h exposure at 25ºC (Navarro, 1977). Since the rate of gas loss is independent of leakage from the enclosure, in well-sealed structures the overall gas loss rate will approach that expected from sorption only.

CONCLUSIONS

Variable pressure tests with N_2, CO_2, PH_3 and MB treated structures, showed that the half life pressure decay-time is a reliable criterion to predict gas loss.

The temperature and barometric pressure determined the intensity of infiltration of O_2 into the cube or the loss of CO_2, PH_3 or MB.

The infiltration rate for O_2 was about 60% greater in the exposed cube as compared to the shaded cube. Gas loss was significantly higher in the exposed cube for CO_2 and PH_3. In contrast, for MB, the loss was significantly higher in the shaded cube.

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